THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Influence of Irradiation on the Properties of Metals

AMER. SOC. TESTING MATERIALS: 'Symposium on Radiation Effects on Materials, Vol. 1.' Sponsored jointly by the American Society for Testing Materials and the Atomic Industrial Forum.

A.S.T.M. Special Tech. Publn. 208, 1957; 190 pp.

The majority of the papers and discussion contained in this volume were presented at the Second Pacific Area National Meeting of the American Society for Testing Materials, in September 1956. Three other contributions, presented at a symposium on radioactive isotopes, are also included in the published volume.

The material is divided into four sections, covering, respectively, the theory of radiation, radiation facilities and the mechanics of testing, experimental tests on fuel and graphite materials, and structural materials (including organics). Individual papers containing reference to nickel-containing materials are indicated below.

L. S. CASTLEMAN: 'Survey of the Effects of Neutron Bombardment on Structural Materials', pp. 108–18.

Includes supporting data on the effect, on yield strength, tensile strength, creep behaviour, brittle-transition temperatures, impact strength and elongation of various materials, among which are nickel, 18–8 chromium-nickel steel and Inconel. The survey is based on material culled from 'unclassified' literature.

D. O. LEESER: 'Radiation Effects on Welds and Notches in Plain Carbon Steels, Stainless Steels and Non-Ferrous Alloys', pp. 154–61.

The programme for the tests reported was designed to establish the effect of irradiation on bend characteristics, impact strength, ductile-to-brittle impact-transition temperatures, and fatigue strength. The paper contains results of tests on chromium-nickel and high-chromium stainless steels, nitrided (Malcomized) chromium-nickel steel, and carbon steel. Reference is also made to tests which were made on nickel, Monel and Inconel alloys, Hastelloy C, precipitation-hardening stainless steels, cobalt-base alloys, and zirconium.

W. F. MURPHY and S. H. PAINE: 'Fast Neutron Effects on Tensile and Hardness Properties of Type 347 Stainless Steel', pp. 162–72.

Report of study of changes in tensile strength and hardness, as a result of irradiation, in annealed and in 70 per cent. cold-drawn niobium-stabilized austenitic chromium-nickel steel. During irradiation the specimens were in contact with a liquid alloy of sodium and potassium, at temperatures between 228° and 316°C.

J. C. WILSON and R. G. BERGGREN: 'Will A.S.T.M. Standards be Influenced by Radiation Effects in Metals ?' pp. 175–82.

The problems associated with estimation of the properties of materials subjected to irradiation is discussed in relation to provisions which could be embodied in specifications. The aim of the paper is to present some preliminary information which may be of service in this connexion, and some suggestions are made as to aid which the A.S.T.M. can give in assembling data which will assist in drawing up sound specifications for materials used in this new field of application. *Inter alia*, the authors present some data on typical radiation effects in austenitic stainless steels.

A. BOLTAX: 'Effects of Radiation Damage on Precipitation-Hardening Alloys, with Special Reference to Copper-Iron Alloys', pp. 183–90.

Studies of the effects of neutron irradiation, on precipitation-hardening alloys, have been carried out on a large number of alloy systems: most of the investigations have been concerned with radiation-induced precipitation from supersaturated solid solutions. 'Irradiation ageing' is reported to have been observed in copper-beryllium alloys at room temperature and in nickel-beryllium alloys at 300°C. (*Jnl. Applied Physics*, 1956, vol. 27, pp. 40–2; *Nickel Bulletin*, 1956, vol. 29, No. 4, p. 64). A summary is made of these data, in comparison with observations made on various other copper-base alloys.

Production of Oxide-free Powders for Powder-Metallurgy Use

See abstract on p. 28.

Grain Growth in Metals

See abstract on p. 2.

Determination of Trace Amounts of Metals in Crops

See abstract on p. 3.

X-Ray Spectrographic Analysis of Alloys

See abstract on p. 28.

Automatic Direct-Reading Spectrograph

'Read Spectrograph Directly for Rapid Analysis.'
Iron Age, 1957, vol. 180, Sept. 26, pp. 124–5.

The 'Automatic Spectro Lecteur' described in the article is designed and produced by Compagnie Radio-Cinéma, Paris. The instrument is claimed to be accurate to ± 1 per cent. Results are read directly from a measuring device and the visual presentation permits both qualitative and quantitative analysis of the sample. Under routine conditions the average time for complete analysis of an alloy is stated to be 1 minute 30 seconds. Change of alloy sample can be carried out in less than 1 minute where the alloys are of the same base, and in less than 5 minutes if the basis metals are different. The article includes illustrated notes on the principal components of the spectrograph, and its operation is described.

The method is applicable to steels and many non-ferrous alloys and provides, *inter alia*, for determination of nickel in steels and in aluminium-, magnesium-, copper-, and lead-base alloys.

Production of Holes in Refractory Alloys by Spark Erosion

See abstract on p. 18.

Stress Relief in Weldments

E. R. PARKER: 'Stress Relieving of Weldments.'
Welding Jnl., 1957, vol. 36, Oct., pp. 433S–40S.

Report sponsored by the Welding Research Council.

A summary is made of current knowledge relating to the origin, nature and effect of residual stresses, and of the influence of thermal and mechanical stress-relieving treatments on the service performance of welded structures.

Shielding Gases for Use in Welding Nickel-containing Materials

See abstract on p. 29.

NICKEL

Reactions of Nickel Sulphide during Roasting

I. KUSHIMA and N. ASANO: 'Roasting of Nickel Sulphide.'
Nippon Kogyo Kaishi, 1957, vol. 73, pp. 103–8.

Observations made of reactions occurring on heating nickel sulphide in an oxidizing atmosphere at various temperatures, are summarized in *Chemical Abstracts*, 1957, vol. 51, Sept. 25, pp. 1313–15.

Preparation of Specially-High-Purity Nickel

K. M. OLSEN: 'Superpurity Nickel Melted under Controlled Atmospheres.'
Metal Progress, 1957, vol. 72, Sept., pp. 105–9.

In connexion with vacuum-tube 'repeaters' for trans-oceanic cables a need arose for nickel of closely controlled composition and specially high purity. This article describes procedure developed by Bell Telephone Laboratories, Inc., to meet these requirements.

Survey of commercially available high-purity nickel resulted in the choice of Mond carbonyl nickel powder as the starting material. The paper describes melting and casting procedure by which, from this high-purity powder, strip of the following composition was produced: iron < 0·005, silicon < 0·005, copper < 0·001, magnesium < 0·005, carbon 0·001, sulphur < 0·001, oxygen 0·0015, hydrogen 0·0001, nitrogen 0·0001, per cent. The procedure involves melting sintered slugs of the carbonyl nickel powder in magnesia crucibles, in a cycle using hydrogen, vacuum and helium atmospheres, with subsequent hot- and cold-rolling under conditions involving no pick-up of impurities.

An appraisal of the purity of the material so produced is made by comparison of its composition (see above) with that of other specially-high-purity nickel for which information has been given in the literature. The low resistivity and high coefficient of the nickel give evidence that it is of a degree of purity not previously obtainable in fair-sized ingots prepared by pyrometallurgical methods. Particular attention is called to the strongly deoxidizing effects of magnesium and aluminium, studied in the course of production of the strip material. As little as 0·003 per cent. of magnesium begins to have an effect and 0·03 per cent. of that element removes practically all the oxygen. A similar effect is observed with additions of aluminium.

The process described has the additional advantage of being applicable also to the production of high-purity nickel alloys. The authors point out the numerous purposes for which such high-purity material would be useful.

Grain Growth in High-Purity Nickel

P. FELTHAM: 'Grain Growth in Metals, with Special Reference to High-Purity Nickel.'
Jnl. Inst. Metals, 1957–58, vol. 86, Oct., pp. 95–7.

The author recently proposed a statistical theory of grain growth in metals: see *Acta Metallurgica*, 1957, vol. 5, p. 97. Verification of the validity of the theory, on the basis of available experimental data on grain growth, is difficult, since most of the published work is invalidated by non-compliance with one or more of several requirements critical for ensuring correct results. The primary object of the work now described was, therefore, investigation of the validity of the theoretically derived law, paying due attention to known potential sources of error. Grain growth in 99·99 per cent. nickel

was studied in the range 700°–900°C., and the following conclusions were drawn:

'The initial and instantaneous mean grain diameters, D_0 and D, were found to be related to the time, t, of isothermal annealing by the expression $D^2 - D_0^2 = Kt$, where $K = K_0 \exp(-H/kT)$. The functional form of this relation, as well as the magnitudes of the parameter K_0 (=0·8 cm.²/sec.) and of the activation energy H (=32 k.cal./g.-atom), are as required by the author's generalized statistical theory of grain growth.'

Influence of Irradiation on the Properties of Nickel

See abstract on p. 1.

Fatigue Behaviour of Nickel

See abstract on p. 7.

Chemisorption of Hydrogen on Nickel

E. L. LEE, J. A. SABATKA and P. W. SELWOOD: 'The Mechanism of Chemisorption : Hydrogen on Nickel. II.'

Jnl. Amer. Chemical Soc., 1957, vol. 79, Oct., pp. 5391–7.

Following investigations reported *ibid.*, 1956, vol. 78, p. 3893: *Nickel Bulletin*, 1956, vol. 29, Nos. 10–11, p. 181, further measurements have been made on supported nickel catalysts of various types, with the aim of further studying the mechanism of chemisorption through the use of magnetism-volume adsorption isotherms. The authors describe four series of experiments: (1) exploratory tests on the effect of changing experimental conditions, (2) determination of the differential heats of chemisorption, (3) a study of the 'slow' (activated) sorption on hydrogen which follows the initial rapid chemisorption, and (4) a further study of the 'hydride' effect, i.e., the reversal of electron transfer which is observed at low temperatures.

The results indicate that no simple relation exists between magnetic change and the type of surface bond found in the samples examined. The slow (activated) process is found to be a true chemisorption, the mechanism of which is similar to the rapid reaction which it follows, except that the slow effect takes place on the smaller particles of the nickel. Other experiments confirmed previous findings that hydrogen adsorbed on these particles at room temperature gives a small positive change of magnetization when the latter is measured at low temperatures.

Hydrogen Overpotential on Electrodeposited Nickel

I. A. AMMAR and S. A. AWAD: 'Hydrogen Overpotential on Electrodeposited Nickel in NaOH Solutions.'

Jnl. Electrochemical Soc., 1957, vol. 104, Nov., pp. 686–9.

Hydrogen overpotential was measured for electrodeposited nickel in NaOH solutions 0·01–0·10*N*, under various conditions. Comparison of the results with those obtained on nickel in the form of wire shows considerable differences.

Determination of Trace Amounts of Nickel in Crops

'Determining Trace Metals in Crops by Chromatography.'

Chemical Age, 1957, vol. 78, Oct. 12, pp. 596–7.

Brief report of paper presented to the Society for Analytical Chemistry.

The author gives details of a rapid, simple and economical chromatographic technique suitable for routine determination of trace amounts of copper, cobalt, manganese, molybdenum, nickel and zinc. The basic procedure used is described as follows:

'Ten milligrams of finely-ground sample is ashed at 550°C. Silica is precipitated in crystalline form by twice-repeat evaporation with hydrochloric acid and heating for two hours on a hot plate. The ash is then dissolved in 0·5 ml. of 50 per cent. hydrochloric acid.

'Whatman No. 1 Pattern C.R.L. slotted paper is used for the chromatographic separations, making possible the simultaneous treatment of ten separate aliquots. A sample (0·01 ml.) of the solution is applied in an even band about 1 cm. from the bottom of the paper strip, which is then formed into a cylinder, clipped together and placed in a tall-form 600 ml. beaker. The beaker is floated in boiling water for 3 minutes, to ensure optimum drying.

'Copper, cobalt and nickel are determined in a 0·02-ml. portion by upward-flow chromatography, using as solvent a mixture of ethyl methyl ketone, hydrochloric acid and water, in the ratio 15:3:2. Development is by means of a 0·1 per cent. solution of rubeanic acid. The presence of nickel is revealed by the appearance of purple-blue bands at the bottom of the paper, cobalt by yellow bands in the middle, and copper by olive-green bands at the top. The coloured bands are visually matched against standard papers, the convenient range being 0·2–2 μg.'

It is stated that if suitable aliquots are used the results are reproducible to within ± 5 per cent. Procedures for determination of molybdenum, zinc, boron and manganese are also given.

Cyanide Titration of Nickel in Presence of Zinc

R. S. YOUNG: 'Effect of Zinc on the Cyanide Titration of Nickel.'

Chemist-Analyst, 1957, vol. 46, Sept., pp. 69, 75.

Report of tests made to evaluate the extent of the interference of zinc and the effect, on such interference, of inhibiting media. Two methods were adopted: (*a*) addition of citrate before the sample solution was made ammoniacal, and (*b*) corresponding additions of sodium pyrophosphate. Increasing amounts of citrate or pyrophosphate were added with increasing amounts of zinc present.

Data on a number of analyses indicate that addition of citrate gives rise to high results for nickel in the presence of zinc, but if the quantity of zinc is less than one-half that of the nickel this error is not significant for routine purposes. Substitution of pyrophosphate for citrate lessens the interference by zinc. With that medium, even when zinc is

present in amounts five times as large as the nickel content, the results, although consistently slightly high, are still within limits acceptable for routine purposes.

Rare-Earth Nickel Oxides

A. WOLD, B. POST and E. BANKS: 'Rare-Earth Nickel Oxides.'

Jnl. Amer. Chemical Soc., 1957, vol. 79, Sept. 20, pp. 4911–13.

Study of the reactions of nickel oxide with the oxides of lanthanum, neodymium, samarium, gadolinium and yttrium, and with a mixture of the oxides of lanthanum and neodymium.

Ammines of Nickel Cyanide

E. E. AYNSLEY and W. A. CAMPBELL: 'The Ammines of Nickel(II) Cyanide.'

Jnl. Chemical Soc., 1957, Sept., pp. 4137–8.

Notes on contradictory findings reported in the literature relating to the ammines of nickel cyanide, followed by report of experimental work in which it was observed that nickel cyanide forms two ammines:

(1) Unstable $Ni(CN)_2$, $4NH_3$, $2H_2O$, and

(2) Stable $Ni(CN)_2$, NH_3, H_2O.

Methods of preparation and characteristics of the compounds are described.

ELECTRODEPOSITION AND OTHER COATING METHODS

Electrodeposition of Nickel from Pyrophosphate Solutions

T. L. R. CHAR: 'Electroplating from the Pyrophosphate Bath. I. Electrodeposition of Single Metals.'

Electroplating and Metal Finishing, 1957, vol. 10, Nov., pp. 347-9.

The paper opens with a condensed review of the literature relating to the use of pyrophosphate solutions, which merit consideration as electrolytes on account of their solubility, non-poisonous nature, stability, and low metal-ion concentration resulting from complex formation. Conditions for the use of such solutions and their practical interest for the electroplating industry are being systematically examined in the laboratories of the Indian Institute of Science, Bangalore. Results of studies on the deposition of alloys have already been published (*Jnl. Scientific and Industrial Research, India, Sect. A*, 1957, vol. 16, No. 7, pp. 324–5; see *Nickel Bulletin*, 1957, vol. 30, No. 12, p. 218). The present communication covers electrodeposition of single metals (tin, zinc, copper, nickel, lead): the material presented is drawn from both published and unpublished data. The solutions used in the author's experiments were prepared by dissolving metal pyrophosphates (stannous, copper or zinc) in sodium or potassium pyrophosphate, and by the addition of alkali pyrophosphate to nickel chloride or lead nitrate. In electrodeposition of nickel it was found necessary to have the chloride ion present: without it the efficiency of the nickel anode was practically zero. Ammonium citrate increased the cathode efficiency. The addition agents studied were chosen on the basis of their performance in well-known plating solutions.

It was found that the pyrophosphate solutions gave satisfactory deposits over a wide range of operating conditions. Cathode and anode efficiencies were close to 100 per cent. The deposits are reported to be adherent, smooth, fine-grained, and non-porous, and to have proved resistant to corrosion in the laboratory atmosphere for 6/13-month periods.

Among the addition agents with which tests were made, several had a brightening effect; details of those suitable for the respective solutions are listed. In nickel plating brightening was obtained by additions of cobalt chloride, sodium sulphite, coumarin, and alpha-nitroso beta-naphthol. The following recommendations are made with regard to optimum conditions for deposition of nickel from pyrophosphate solutions.

Composition, g./L.	Nickel 29·4 P_2O_7 (K) 234·8 Ammonium citrate 33·3
pH	9·5
Temp., °C.	60
Bath voltage ..	1·5-2·6
Specific resistivity, ohm/cm.^{3}	4·51
Agitation	—
Cathode	Cu
Current density, cathode amp./dm^2 (max.)	6·0
„ „ anode amp./dm.^{2} (max.)	8·0
Cathode efficiency, % ..	86-93
Anode efficiency, % ..	96-97
Cathode polarization, volts	0·47-0·81
Throwing power, %	22*

Comparison of the performance of such electrolytes with that of Watts-type solutions indicates that the former have the better throwing power.

Adhesion of Nickel Coatings as Affected by the Silicon Content of Steel

J. J. DALE and H. K. LUTWAK: 'The Effect of Silicon in a Low-Alloy Steel on the Adhesion of Heavy Nickel Plating.'

Trans. Inst. Metal Finishing, 1957, vol. 34, pp. 133–8; *Bull. Inst. Metal Finishing*, 1957, vol. 7, No. 2, *Summer Issue*.

In the re-building of worn or over-machined components by means of heavy nickel coatings, prerequisites for satisfactory adhesion of the deposits to the basis metal are effective cleaning of the surface of the metal prior to plating, and removal, by etching,

* Calculated by the method described by GARDAM (*Trans. Faraday Soc.*, 1938, vol. 34, pp. 698-711.)

of surface deformation produced by machining or polishing. The etching methods used vary with the basis metal, and in the case of steel components the presence of alloying elements calls for consideration, since some of these are insoluble in normal etching solutions. Carbon formerly caused difficulty, because, after etching, it remained as an insoluble dark film ('smut') on the surface of the steel, inhibiting adhesion, but this problem has been practically solved by anodic treatment in sulphuric-acid solution, under closely controlled conditions. In this connexion reference is made to fundamental investigations carried out at the Armament Research Establishment (previously R.D. Woolwich), the results of which were published in *A.R.D Electrodeposition Memorandum No.* 6, 1943.

The present paper is concerned with a rebuilt component which had been prepared for plating in accordance with recommended procedure, but which failed by separation of the nickel deposit from the basis metal during machining. The steel concerned was of the B.S.En 25 type (carbon 0·27–0·35, silicon 0·10–0·35, manganese 0·50–0·70, sulphur 0·050, phosphorus 0·050, nickel 2·30–2·80, chromium 0·50–0·80, molybdenum 0·40–0·70, per cent.). Investigation of the source of the 'smut' film which persisted on the steel, and of consequent poor adhesion, focused attention on the silicon content of the steel. It was assumed that silicon, which is oxidized to silicic acid during anodic solution of steel in weak acids, would also be oxidized if strong acids were used as electrolytes. It was then postulated that the silicic acid, which would be insoluble in the electrolyte, would form, on the surface of the steel, an adherent film impervious to a normal anodic etch. If this film were of low carbide content it would be invisible. The abnormally high silicon content of the steel of the component in question (0·37 per cent.) tended to support this theory, and treatment of the steel with warm 1 per cent. sodium-hydroxide solution (in which silicic acid is readily soluble) corroborated the presumption. Such treatment freed the surface from 'smut', and there was consistently good adhesion of the nickel deposits to the steel so prepared. Experiments carried out to determine the critical silicon content indicated that if it is more than about 0·25 per cent. the conventional anodic etch is likely to be unsatisfactory and treatment in sodium-hydroxide solution is advisable.

Although the authors' hypothesis with regard to the rôle of silicon is not yet definitely proved, a number of full-scale repairs by the new technique have given consistently good adhesion on steel of the type discussed. The following procedure is recommended as a general guide, but it is emphasized that each stage should be considered in the light of specific applications.

(1) Degrease in trichlorethylene vapour.
(2) Cathodic clean in alkali.
e.g., 8 oz./gal. NaOH; 8 oz./gal. Na_2CO_3.
(3) Anodic clean in alkali (in a separate tank).
(4) Cool to 12°–15°C. with crushed ice.
(5) Anodic etch in 30% sulphuric-acid solution at 30 amp./sq. ft. for 15 min.; temperature not above 15°C.
(6) Increase current density to 1000 amp./sq. ft. for 15 sec.
(7) Rinse, scour with pumice and rinse again.
(8) Anodic etch in fresh 30% sulphuric-acid solution at 1000 amp./sq. ft. for 15 sec.; temperature not above 15°C.
(9) Rinse with cold and then with hot water.
(10) Immerse in fresh 1% sodium-hydroxide solution at 50°C. and brush for several minutes.
(11) Rinse.
(12) Inspect for presence of 'smut' patches.

If the surface is unsatisfactory repeat operations 6-11.

(13) Rinse with dilute (app. 0·3%) sulphuric-acid solution.
(14) Nickel plate.
(15) Anneal, after plating, for 2 hours at 200°C. in oil or in air.

Hydrogen Overpotential on Electrodeposited Nickel

See abstract on p. 3.

'Oil Pores' in Electrodeposited Nickel Coatings

K. SOMMER: 'Oil Pores in Electrodeposited Nickel Coatings.'

Metalloberfläche, 1957, vol. 11, Nov., pp. 366–7.

The paper describes a type of localized porosity encountered in nickel coatings, caused by patches of greasy substances on the basis metal. The fact that inadequately cleaned surfaces give rise to faulty adhesion is well recognized, and the author points out that contamination by greasy matter on the surface of the basis metal may also cause porosity of the deposited coating. In the present instance adhesion of the coating was satisfactory, except in the regions of the pores. The 'oil pores' were observed on tubular components which had been treated by the following sequence: electrolytic cathodic degreasing in a hot solution, water rinsing, pickling in 10 per cent. sulphuric acid, water rinsing, plating from a bright-nickel solution, and final chromium coating of work so prepared. Plating normally proceeded satisfactorily, but in some cases oily lubricant or other greasy matter emerged from the interior of the tubes and collected in patches on the surface of the degreasing and pickling baths. After rinsing of the work no oil or other contaminant could be seen on the surface of the tubes, but, on plating, localized porosity was observed in the deposits. The defect was eliminated by removal of all traces of greasy matter from the cleaning and pickling baths. The author urges the need for improved preparatory procedure.

The nature of the oil pores is described by reference to photomicrographs of typical examples, and possible reasons for their peculiar ridged formation are discussed.

Nickel Plating of Lead-base Alloys

'Proposed Tentative Recommended Practice for Preparation of Lead and Lead Alloys for Electroplating.'

Plating, 1957, vol. 44, Oct., pp. 1104-6.

See also A.S.T.M. B319-57T, contained in 1957 *Supplement to Book of A.S.T.M. Standards, Pt.* II, pp. 91-5.

This schedule has been approved by the sponsoring committee of the American Society for Testing Materials, for use pending adoption as standard.

It covers preparatory treatment and plating procedure for lead-base materials of various types, e.g., stereotype metal, alloys for ornamental or decorative ware or for costume jewellery, and small parts which are to be barrel-plated. In addition to general recommendations, the schedule contains full details of cleaning and plating cycles which have proved successful under commercial plating conditions.

Bright-Nickel Plating of Costume Jewellery

E. N. CASTELLANO: 'Bright-Nickel Plating in the Costume-Jewellery Industry.'

Plating, 1957, vol. 44, Oct., pp. 1083-5.

The article emphasizes the importance of bright-nickel plating as a factor in the rapidly growing costume-jewellery industry, and gives some general notes on methods used in plating shops handling that type of work. In connexion with the use of rhodium plating as a final tarnish-resisting coating, the writer urges the advantages of the 'silver' colour obtained by means of the nickel undercoat.

Diffused Nickel-Cadmium Deposits

H. J. WEST: 'Diffused Nickel-Cadmium Deposits.'

Metal Finishing, 1957, vol. 55, Oct., p. 74.

This short article describes a method for production of nickel-cadmium coatings on low-alloy or carbon-steel parts. The coated parts are stated to be suitable for service involving temperatures up to about 900°F. (480°C.).

Brazing and welding should be completed before plating, and components which have a hardness higher than Rockwell C40 or have been ground after heat-treatment should be suitably stress-relieved before coating. Cleaning and preparatory procedure is the same as that generally used before nickel plating. The nickel-plating solution is a chloride or sulphate-chloride type: the composition recommended is:

	oz./gal.
Nickel sulphate	36-40
Nickel chloride	8-16
Boric acid	4

pH	3·5-4·5
Current density	40-60 amp./sq. ft.
Temperature	110°-130°F.(45°-55°C.)
Thickness of deposit	0·0002-0·0004 in.

After nickel plating the parts are rinsed and neutralized in an alkaline solution and immediately cadmium-plated, using a solution of the composition shown in the right-hand column.

	oz./gal.
Cadmium metal	2·5-4·5
Free cyanide	7·0-11·0
Sodium hydroxide	1·0-2·0

Current density	10-15 amp./sq. ft.
Room temperature	
Thickness of deposit	0·0001 in.

In most cases a chromate dip is required after the cadmium-plating stage, to eliminate fingerprint staining, and it is also essential to ensure that the cadmium deposited is never more than half the thickness of the nickel coating. A larger proportion of cadmium will cause 'balling'.

The plated coatings are diffused by treatment at 630°F. (330°C.) for 30 minutes to one hour. It is recommended that the treatment should be begun at about 200°F. (95°C.) and that the temperature should gradually be raised to 630°F. This higher temperature is near the melting point of cadmium and if the part is subjected to this temperature immediately after plating, the cadmium will have a tendency to melt before diffusion can take place, causing 'balling' and the occurrence of small pimples of fused cadmium on the surface. After diffusion the plating should have a dull matt grey-to-black finish.

The acceptance test consists in heating in air at 700°F. (370°C.) for 23 hours and subsequent heating at 1000°F. (540°C.) for one hour, after which there should be no blistering or cracking of significant surfaces. The diffusion-coated parts should also show no corrosion after being subjected to a salt-spray test for 100 hours.

Influence of Cadmium Plating on Properties of Ultra-High-Strength Steels

See abstract on p. 12.

NON-FERROUS ALLOYS

Nickel-Chromium Alloys: Constitution and Structure

See abstract on p. 13.

Nickel-Zirconium System

E. SMITH and R. W. GUARD: 'Investigation of the Nickel-Rich Portion of the System Ni-Zr.'

Trans. Amer. Inst. Mining and Metallurgical Engineers. 1957, vol. 209, pp. 1189-90; *T.N.* 385*E*.

Jnl. of Metals, Oct., 1957.

Investigation of the nickel-rich end of the nickel-zirconium system has been prompted by an interest in the effect of small amounts of zirconium in high-temperature alloys. This paper briefly reports a study of alloys containing up to 50 at. per cent. zirconium, examined in the as-cast condition and also after annealing for six hours at 1175°C. (or 1000°C. for alloys containing between 25 and 45 at. per cent zirconium). The authors present a tentative constitutional diagram for the system up to 50 at. per cent. zirconium, based on the results of microstructural and X-ray observations

coupled with determinations of peritectic and eutectic temperatures. The diagram varies significantly in certain respects from those developed by earlier investigators, to whose work reference is made.

Aluminium-base Alloys containing Iron, Copper and Nickel

G. V. RAYNOR and B. J. WARD: 'Aluminium-Rich Alloys of the Quaternary System Aluminium-Iron-Copper-Nickel.'

Jnl. Inst. Metals, 1957–58, vol. 86, Nov., pp. 135–44.

Report of study of the aluminium-rich alloys of the quaternary system in the solid state at 530°C., by means of metallographic and X-ray diffraction methods and by electrode-potential measurements. The following observations are made:

'In the composition range investigated, the aluminium-rich solid solution may be in equilibrium with $CuAl_2$, with the ternary compound *T*(FeCu), or with the ternary compound *T*(NiCu). By the extrapolation of results obtained at total solute concentrations of 10 and 15 wt.-%, the compound *T*(FeCu) is shown to be capable of dissolving nickel up to a limit, at 530°C., of approximately 6·8 wt.-%. The solubility of iron in *T*(FeCu) is much smaller. Experiments with selected alloys which had been slowly cooled from the melt made possible the preparation of pure samples of *T*(FeCu), *T*(NiCu) and *T*(FeNi) from the appropriate ternary systems and the quaternary system. Chemical analyses confirmed the compositions $FeCu_2Al_7$ and $NiCu_3Al_6$ respectively, for *T*(FeCu) and *T*(NiCu), and showed that, in the former, the iron atoms could be replaced by nickel atoms at least up to a nickel content of 6·5 wt.-%, in good agreement with results in the solid state. The solubility of iron in *T*(NiCu) is small, approximately 0·8 wt.-%. *T*(FeNi) can contain little copper in solid solution.'

Influence of Nickel on Constitution of Copper-Lead Alloys

E. PELZEL: 'Copper-Lead Alloys. Part II. The Influence of Nickel on the Form of the Constitutional Diagram.'

Metall, 1957, vol. 11, Aug., pp. 667–70.

The author discusses earlier literature, with particular reference to apparently contradictory conclusions reached by various investigators, and reports experimental observations which led to re-consideration of the ternary alloy system in the region up to about 10 per cent. nickel.

'Part III. The Specific Volume of Solid and Liquid Alloys and Mode of Solidification.'
Ibid., Nov., pp. 954–8.

Study of the characteristics of binary copper-lead alloys and of lead alloys containing 30, 40 and 50, per cent. CuNi.

Resistance of Metallic Materials to Cavitation-Erosion

See abstract on p. 23.

Fatigue Behaviour of Monel, Nickel and Copper

C. A. STUBBINGTON and P. J. E. FORSYTH: 'Some Metallographic Observations on the Fatigue Behaviour of Copper and Nickel and Certain of their Alloys.'

Jnl. Inst. Metals, 1957–58, vol. 86, Oct., pp. 90–4.

Earlier work by the authors had shown that a 96–4 per cent. aluminium-copper alloy, in the solution-treated condition, exhibited a slip-band-extrusion phenomenon when subjected to cyclic stress, both at room temperature and at temperatures of the order of 250°C. It was considered that this effect was probably due to soft spots of equilibrium concentration formed in the matrix, at the test temperature, by localized over-ageing. To determine the general validity of this assumption, further fatigue tests were carried out on Monel (nickel 68, copper 32, per cent.), an alloy showing complete solubility at the test temperature. Tests were made also on electrolytic copper, on nickel and on a 75–25 copper-nickel alloy.

The specimens, which were fabricated from cold-rolled, vacuum-annealed sheet 0·016-in. thick, were fatigue-tested by vibrating at room temperature and at temperatures of the order of 300°C. Monel was also tested in the cold-worked condition. Photomicrographs illustrate the phenomena observed.

Examination of the Monel specimens revealed the presence, at both test temperatures, of the slip-band-extrusion phenomenon previously observed: the effect was particularly marked on cold-worked specimens fatigued at 300°C. Striations and slip-band phenomena were also observed in the copper, the 75–25 copper-nickel alloy and the nickel. Examples of deformation and cracking in the region of twin boundaries were visible in the nickel and copper.

It is concluded that the investigation has provided further evidence of the existence of localized soft spots in fatigued metals: the presence of the extrusion phenomenon in stable solid solutions is an indication that local over-ageing is not a necessary factor in the process. In these localized soft spots any subsequent dislocation movement is concentrated, leading to an excess vacancy distribution in such zones, and eventual failure. In the case of the annealed pure metals, the soft spots are produced by a hardness differential between a matrix hardened by microslip and zones in which coarse slip movement is occurring. In stable solid solutions the hardness differential may arise from rearrangement of solute atoms. In the annealed Monel specimen fatigued at 300°C. a difference in etching characteristics between regions exhibiting slip-band extrusion and the matrix is believed to be due either to solute-atom migration away from such regions, or to the formation, under the action of cyclic stress, of zones of which the degree of disorder is greater than that of the matrix. Other mechanisms which cause local softening are over-ageing in age-hardening alloys and recrystallization in cold-worked metals and alloys. In most types of material combinations of these mechanisms will be operative.

Sulphide-Corrosion Cracking of Bolts

See abstract on p. 22.

Oxidation Characteristics of a Copper-Nickel Alloy

See abstract on p. 19.

Brittle-to-Ductile Fracture in Chromium-base Alloys

E. P. ABRAHAMSON and N. J. GRANT: 'Brittle-to-Ductile Transition Temperatures of Binary Chromium-base Alloys.'

Trans. Amer. Soc. Metals, 1957, vol. 50, *Preprint* 14: 17 pp.

Report of study of the transition temperatures of ductile-to-brittle fracture in as-cast binary alloys of chromium containing up to 30 wt. per cent. of 21 different elements, in binary combinations. An arbitrary 65° bend angle was used as the measure of the transition temperature. Correlation is made between the rate of transition-temperature change and the electron configuration of the solute element, and a tentative explanation of transition temperatures is advanced.

Evidence is presented to show that, if the transition temperature of the chromium used is sufficiently low, it is possible to produce chromium-base alloys which are ductile at room temperature. Such alloys will fall within the range 60–90 per cent. chromium.

Automatic Direct-Reading Spectrograph

See abstract on p. 2.

Applications of Nickel-base Corrosion-Resistant Alloys

See abstract on p. 24.

Report of the Royal Mint: Nickel-containing Coinage

'Eighty-Seventh Annual Report of the Deputy Master and Comptroller of the Royal Mint for the Year 1956.'

Published by *H.M. Stationery Office*, 1957; 75 pp. + index. Price 4/6.

The report includes the normal statistical information on U.K. silver, cupro-nickel, nickel-brass and bronze coinage, currency withdrawn, assays made, etc. Details of the activities of the associated Mints in Australia are also given. The only coinage of completely new design executed at the Mint during the year 1956 was a cupro-nickel threepenny piece, one of three denominations intended for use in the States of Guernsey. These coins, together with medals and seals struck during the year, are illustrated.

Nickel-containing Aluminium Alloys for Contact with High-Temperature Water

See abstract on p. 19.

Stability of Cyanogen: Monel Containers

See abstract on p. 29.

Shielding Gases for Use in Welding Nickel-containing Materials

See abstract on p. 29.

NICKEL-IRON ALLOYS

Vacuum Heat-Treatment of Nickel-Alloy Springs

T. RAEDY: 'Vacuum Heat-Treating Hairsprings.'

Steel, 1957, vol. 141, Oct. 21, pp. 94, 97.

Hairsprings for watches made by Associated Spring Corporation, Bristol, Conn. are of Ni-Span C (nickel - chromium - iron - titanium precipitation - hardening alloy). The modulus of rigidity of this material is constant over a temperature range of −75° to +220°F. (−60° to +104°C.).

Optimum properties are attained in these alloys only by treatment at high temperatures, and exposure to conventional furnace atmospheres at such temperatures would cause severe oxidation, which would spoil the surface finish essential for precise functioning of the springs and also give rise to trouble in soldering and brazing. Heat-treatment is therefore carried out in vacuum: the procedure used is described in this article. After heat-treatment the springs are left in the evacuated retort while cooling to room temperature.

Nickel and Nickel-Alloy Waveguides

J. ALLISON, F. A. BENSON and M. S. SEAMAN: 'Characteristics of Some Ferrous and Non-Ferrous Waveguides at 27 Gc/s.'

Proc. Instn. Electrical Engineers, Pt. B, 1957, vol. 104, Nov., pp. 599–602.

Losses in waveguides and cavity resonators, produced by the flow of induced currents in the metal walls, can be calculated from formulae derived on the assumption that the surfaces are perfectly smooth. It is, however, well known that at micro-wave frequencies the measured losses are considerably greater than those calculated from such formulae, and it has been concluded, from a series of investigations, at frequencies about 10 Gc/s, on drawn copper, brass and electroplated waveguides, that such discrepancies are due solely to surface roughness. The attenuations produced by waveguides of nickel, mild-steel, Mumetal, Radiometal and Rhometal have also been determined in the frequency range 9·9·675 Gc/s, and it has been shown that such measurements enable the effective high-frequency permeabilities of the materials to be estimated.

This paper reports measurements of the attenuations produced by air-filled rectangular waveguides of brass, copper, aluminium, nickel silver, nickel, mild steel, Radiometal*, Rhometal* and Mumetal*, at a frequency of 27 Gc/s. From the results of these measurements the effective permeabilities of the materials were estimated.

* Nickel-iron-base alloys containing, respectively, about 49, 37·5 and 72 per cent. of nickel.

CAST IRON

Properties of Alloy Cast Irons

J. H. CULLING and J. F. WALLACE: 'Alloy Cast Irons Can Solve Tough Problems.'

Materials in Design Engineering, 1957, vol. 46, Oct., pp. 140–4.

The article is adapted from a section prepared for inclusion in the Iron Castings Handbook, to be published by the Gray Iron Founders' Society. A general review is made of the range of properties obtainable in cast irons by use of various alloying additions (chiefly nickel, chromium and silicon). The subject is divided into four sections, dealing respectively with corrosion-resistant irons, heat-resistant irons, special-purpose irons and high-alloy ductile irons. In each section the properties of the irons are evaluated in relation to the specific applications for which they have been developed: the information given in the text is supplemented by tabular data on physical and mechanical properties. Emphasis is placed throughout on the value of nickel and other alloying elements as a means of securing practically useful properties and of widening the sphere of application of cast iron.

German, British, French and American Standards for Grey-Iron Castings

'Comparison of American, English, French and German Standards for Grey- and Malleable-Iron Castings.'

Giesserei, 1957, vol. 44, Oct. 10, pp. 642–4.

Six tables show the German specifications, with the relevant British (B.S.), French (A.F.N.O.R.) and American (A.S.T.M.) standards.

Resistance of Cast Iron to Cavitation-Erosion

See abstract on p. 23.

CONSTRUCTIONAL STEELS

British and U.S. Steel Specifications

W. B. KEMMISH: 'A Comparison of British and American Standard Steels.'

Materials in Design Engineering, 1957, vol. 46, Oct., pp. 171, 173, 175, 177; Nov., pp. 167, 169, 171.

This article is closely similar to that published in *Machinery (London)*, 1956, vol. 89, July 20, pp. 167–9 (see *Nickel Bulletin*, 1956, vol. 29, Nos. 8–9, pp. 167–9). The author outlines the basic differences in methods of specifying employed in the U.K. and the U.S.A., and presents a tabular summary of specifications of the B.S.En and S.A.E./A.I.S.I. series. In this second article the types covered have been extended to include manganese-nickel-chromium constructional steels, spring steels and B.S.En and A.I.S.I. corrosion- and heat-resisting types.

Martensitic Transformation in the Nickel-Iron System

L. KAUFMAN and M. COHEN: 'The Martensitic Transformation in the Nickel-Iron System.'

Trans. Amer. Inst. Mining and Metallurgical Engineers, 1956, vol. 206, pp. 1393–401.
See *Nickel Bulletin* 1957, vol. 30, No. 1, p. 7.

Discussion on this paper (*Trans. Amer. Inst. Mining and Metallurgical Engineers*, 1957, vol. 209, pp. 1314–15; *Jnl. of Metals*, Oct., 1957) includes a contribution from A. G. HAYNES summarizing data (assembled from the literature and from experimental work) on martensite-transformation ranges for a variety of nickel-containing steels. In the light of these data, comments are made on the authors' conclusions.

Authors' reply, p. 1315.

Effect of Hydrogen upon Martensite Formation in Steels

R. KUMAR and A. G. QUARRELL: 'The Effect of Hydrogen upon Martensite Formation in Low-Alloy Steels.'

Jnl. Iron and Steel Inst., 1957, vol. 187, Nov., pp. 195–204.

It is now generally accepted that hairline cracks in steels of high hardenability form in the presence of hydrogen if decomposition of austenite is not complete above the temperature at which the martensitic transformation begins, and that a mixed structure containing small amounts of martensite is most prone to flaking and to cracking during welding. There is, however, some controversy (as the authors indicate by reference to the literature) as to the extent to which the formation of hairline cracks is attributable to transformation stresses. Many of the characteristics of hairline-crack formation strongly suggest that an important part may be played by these stresses, and by the effect of hydrogen upon them, but relatively little work has been done on the influence of hydrogen upon transformation. The present investigation was undertaken to add to the somewhat sparse data on this aspect.

The steels studied (various grades of low-alloy nickel-chromium, nickel-chromium-molybdenum, chromium, and chromium-molybdenum types, and high-chromium stainless steels) were selected to ensure that no transformation occurred either during quenching to the M_s-M_f range or during tempering. Full details are given of the experimental conditions adopted as suitable for the respective steels. The specimens were subjected to the following treatment:

(1) Austenitized in hydrogen or nitrogen.

(2) Quenched in a liquid bath to a temperature in the M_s-M_f range and held there for a time

sufficient to equalize the temperature across the specimen.

(3) Immediately tempered in hydrogen or nitrogen (at temperatures and for times suitable for the respective steels), followed by quenching in water.

(4) Immediately slit into two parts for—

(*a*) determination of the hydrogen content, with a minimum of delay, in a low-temperature (650°C.) vacuum-extraction apparatus;

(*b*) metallographic examination.

In the first stage of the investigation martensite formation in hydrogen-treated and nitrogen-treated specimens was studied over the entire martensite range. The investigation covered also the effect of carbon content in two 3 per cent. chromium-molybdenum steels (carbon 0·86 and 0·57 per cent., respectively). The comprehensive data derived from the tests are presented in tabular and graphical form. The conclusions drawn are summarized as follows:

'(*a*) Hydrogen depresses to lower temperatures the later stages of the transformation in

0·30% carbon, 3% nickel-chromium steel to En 23.
0·29% carbon, 4½% nickel-chromium-molybdenum steel to En 30B.
free-cutting 13% chromium steel.
13% chromium steel to En 56A.
carbon-chromium steel to En 31.

'(*b*) Hydrogen has no effect on the transformation in

0·29% carbon, 3% chromium-molybdenum steel to En 40.
0·22 carbon, 3% chromium-molybdenum steel
0·55% carbon, 4½% nickel-chromium-molybdenum steel.
0·80% carbon, 4½% nickel-chromium-molybdenum steel.

'(*c*) Hydrogen depresses the M_S temperature, and causes the later stages of the transformation to occur at higher temperatures, in

0·57% carbon, 3% chromium-molybdenum steel.
0·86% carbon, 3% chromium-molybdenum steel.'

The authors consider that their results are too few to justify sweeping generalization, but that it would appear that the effect of increasing the carbon content of the 3 per cent. chromium-molybdenum and 4½ per cent. nickel-chromium-molybdenum steels is to modify the effect of hydrogen in the direction (*a*)→(*b*)→(*c*). A tentative explanation is advanced to account for the carbon effect. It is suggested that there is a strong interaction between the hydrogen and carbon atoms, which tends to prevent clustering of the latter, and so reduces the possibility of occurrence of carbon-free regions of austenite which could serve as embryos for martensite transformation.

The significance of the stabilization of austenite by hydrogen and the occurrence of 'burst' formation of martensite is discussed in relation to hairline cracking. It is argued that transformation of austenite must lead to tensile stresses in the matrix, and that retention of austenite to a lower temperature will increase the tensile stresses. These stresses, superimposed on the existing stress pattern in steel, will be sufficient to disrupt a steel embrittled by hydrogen. The hydrogen-pressure effect would then play an important part in extending the cracks.

The authors consider that the experimental observations made indicate clearly that hydrogen can exert a profound effect upon transformations in steel and that there is an urgent need for further work on this subject.

Inter-Relation of Tempered-Martensite Content and Endurance Limit in Steels

F. BORIK, R. D. CHAPMAN and W. E. JOMINY: 'The Effect of Per Cent. Tempered Martensite on Endurance Limit.'

Trans. Amer. Soc. Metals, 1957, vol. 50, *Preprint* 16; 24 pp.

A comparatively small proportion of the literature correlating martensitic structure with mechanical properties has reference to endurance characteristics of steels. The work reported in this paper was done to supplement data on this aspect of structure/property relationships.

The tests were made on five low-alloy steels containing 0·39–0·45 per cent. carbon, representative of types showing varying hardenability: the group included a nickel-chromium S.A.E. 4340 type and a boron-treated lean triple-alloy grade. By using various quenching media and by adjustment of cross section of the quenched bars, varying amounts of martensite were produced by continuous cooling (a method chosen in preference to isothermal transformation because of its industrial interest).

It was found that in the steels as quenched and tempered to Rockwell C36, a relationship exists between percentage of martensite formed on quenching and the endurance limit. It is further demonstrated that a small percentage of non-martensitic structure has a deleterious effect on the fatigue strength of the steels tested. Down to 85 per cent. martensite the effect is relatively rapid, but below that percentage the endurance limit is affected very little by microstructure.

Elevated-Temperature Properties of Constructional Alloy Steels

A. B. WILDER, E. F. KETTERER and D. B. COLLYER: 'Stability of A.I.S.I. Steels.'

Trans. Amer. Inst. Mining and Metallurgical Engineers, 1957, vol. 209, pp. 1176–81; *T.P.* 4503*E*. *Jnl. of Metals*, Oct., 1957.

The A.I.S.I./S.A.E. constructional-steel series (Ni, Ni-Cr, Cr-Mo, Ni-Cr-Mo, Ni-Mo and Cr-V types), although widely used in the automotive and other engineering industries, have not up to the present been generally adopted for elevated-temperature service. The purpose of the investigation now reported was the study of changes in microstructure and mechanical properties occurring in representative steels of these types after prolonged exposure to elevated temperatures. Each steel was tested at

two carbon levels, in the normalized or annealed state, and in the welded condition, after exposure for 10,000 and 34,000 hours at 900° and 1050°F. (482° and 565°C.) and, in some cases, at 1200°F. (650°C.). The work was one of a series of investigations which have developed since 1943, when the significance of graphitization in steels on prolonged exposure to elevated temperatures was first fully appreciated (see *Trans. Amer. Soc. Mechanical Engineers*, 1944, vol. 66, p. 5; *ibid.*, 1946, vol. 68, p. 597). In addition to evaluation of the structural stability of the steels after the long-time high-temperature exposure, tensile and creep tests were made after 10,000-hours' heating at the three temperatures. The behaviour of the steels was closely compared with that characteristic of the types of chromium-molybdenum steel currently used for piping in steam plant.

A detailed report is made on the graphitizing characteristics of the respective materials, structural changes are illustrated, and comparison is made of tensile properties, creep-rupture strength and ductility after the prolonged heating.

The results demonstrate the structure-stabilizing influence of chromium and its beneficial effect on creep-rupture strength. It is concluded that the chromium-molybdenum steels now used for elevated-temperature piping service are more suitable for such applications than the other constructional steels included in these tests.

Mechanical Properties of Nickel-Alloy Steels

MOND NICKEL CO., LTD.: 'The Mechanical Properties of Nickel-Alloy Steels.'

Publn. 468D*, 1957; 95 pp.

This is a revised and amplified edition of a publication first issued in 1951. It is intended to serve as a reference handbook for engineers, designers, metallurgists and heat-treatment superintendents concerned with production and utilization of constructional steels.

The mechanical properties and hardness of representative nickel-containing heat-treatable and case-hardening steels (selected from British Standard 970: 1955) are detailed in a comprehensive series of tables and figures. The information given covers specification requirements for chemical compositions, heat-treatment and mechanical properties, and representative test data incorporate the effect of size on properties (mass effect). For the direct-hardening steels tempering diagrams are included.

Both sections of the handbook have been amplified, in the revised edition, to include some steels which have been introduced and/or become established since the issue of the original publication.

Appendices contain the following further information relevant to the use of the steels covered by the main text:

(1) Miscellaneous Mechanical Properties
(Case hardness: torsion; fatigue; physical properties of a 3 per cent. nickel-chromium steel of B.S. En 23 type)

(2) Heat-treated Nickel-Alloy Steels for Aircraft
(The 'S' series of steels, compared with their counterparts in the B.S. En Series)

(3) Automobile Nickel-Alloy Steels, as specified in 'British Standards for the Automobile Industry', B.S. Handbook No. 8, 1950.

(4) Ruling Section. Conversion of Rectangular Sections and Plates into Equivalent Rounds.

(5) Comparison of En Steels with other Official Specifications operative in the U.K., U.S.A. and European countries.

(6) Comparison of Various Official Tensile and Impact Test Pieces (based on conditions laid down by specifications in the U.K., various European countries and the U.S.A.)

(7) Hardness and Strength Conversions.

* *We shall be pleased to supply a free copy of this publication.*

Influence of Alloying on Temper-Brittleness of Nickel-Alloy Steels

M. P. BRAUN: 'Effect of Type of Alloying on Temper-Brittleness of Chromium-Manganese-Nickel Steel.'

Metallovedenie i Obrabotka Metal, 1957, No. 1, pp. 38–41.

The steels studied contained carbon 0·30–0·40, chromium 1–1·2, manganese 1–1·25, per cent. Fourteen steels had nickel contents between 1·5 and 1·7 per cent. and the ten remaining specimens contained 2·2–2·5 per cent. nickel. The following further alloy elements were added, singly or in combination, in amounts up to 3 per cent.: vanadium, molybdenum, titanium, tungsten, niobium, aluminium. Heat-treatment consisted in quenching from the hardening temperatures usually employed for such steels, and from 1000°C., followed by tempering in the range 200°–650°C. After tempering at 500°–650°C. some specimens were furnace-cooled (0·4°/min.) and some were water-quenched.

A summary of the results of the tests is given in *Chemical Abstracts*, 1957, vol. 51, Sept. 25, p. 13,709.

Temper-Embrittlement: Influence of Carbon Content

E. B. MIKUS and C. A. SIEBERT: 'A Study of the Rôle of Carbon in Temper-Embrittlement.'

Trans. Amer. Soc. Metals, 1957, vol. 50, *Preprint* 39; 19 pp.

The authors open their paper by clearly defining the term 'temper-embrittlement' as covering the adverse effect, on impact properties, resulting from slow cooling through the temperature region 1250°–700°F. (675°–370°C.) or from isothermal holding in that range for an extended period. The many studies of this phenomenon and the many experimental results obtained have not yet resulted in an agreed theory which will adequately explain the observations made. Many investigators have, however, advanced theories according to which carbon atoms or carbide particles are a significant factor, and the aim of the work now reported was to examine this aspect

more fully, by studying the effect of carbon in a steel known to be susceptible to temper-embrittlement (carbon 0·39, silicon 0·28, manganese 0·78, sulphur 0·019, phosphorus 0·010, nickel 1·26, chromium 0·61, molybdenum 0·04, per cent.). Examination was made of the steel in a severely embrittled state and in the tough condition, the rôle of carbon being evaluated by determining the distribution of the carbon atoms in the steel in the two conditions. Electron microscopy was used to study the size, shape and distribution of the carbide particles, and a stripping-film autoradiographic technique was also employed, using carbon-14. The account of the experimental work is preceded by a concise review of the literature relating to the influence of alloy elements on temper-embrittlement, and on the theories which have been put forward to explain the phenomenon.

The observations made on the tough and severely-embrittled steel specimens lead to the following conclusions with reference to the distribution and behaviour of carbon:

'No continuous film of any kind is observed in the prior austenite grain boundaries of the temper-embrittled structure developed in the steel examined. Although carbides are observed in the prior austenite grain boundaries of the embrittled structure, it is considered unlikely that these have any effect on temper-embrittlement, since carbides were observed also in similar positions in the structure of the steel in the tough condition.

'The prior austenite grain boundaries, as revealed by an ethereal picric etch, are grooves between the grains. They increase with time of etching and degree of embrittlement, and are not a staining effect of the etchant. Autoradiographic studies (using carbon-14) and electron microscopical observations of the embrittled structures did not reveal any segregation of carbon atoms or carbides to the prior austenite grain boundaries.'

It is posited that temper-embrittlement results from a strain developed across prior austenite grain boundaries at the embrittling temperature, as a result of the contraction of the ferrite lattice when dissolved alloying elements are removed from solution in it by a reaction with the existing minor carbide phases.

Pre-Stressing of Chromium-Nickel-Molybdenum-Steel Torsion-Bar Springs

N. E. HENDRICKSON: 'Increasing Strength and Fatigue Life of Steel Stressed in Torsion.'

A.S.T.M. Bulletin, 1957, Sept., pp. 40–2.

Among the processes developed in the last 25 years to improve the fatigue life of highly stressed components, shot-peening and pre-stressing are becoming increasingly important. Pre-stressing is especially valuable if the component is to be subjected in service to loads of considerable magnitude applied in one direction only and it is thus of significance in relation to vehicle suspension springs. On torsion-bar springs for track-type vehicles, which are the subject of this paper, shot-peening and pre-stressing have been so effectively applied that the springs can operate without breaking or taking excessive set through a stress range of approximately 125,000 p.s.i. (56 tons per sq. in.) with maximum stress at a value which is that of the elastic limit of the steel prior to the mechanical treatment. Such treatment reduces by half the amount of steel required to ensure a good fatigue life.

The author describes the production of torsion-bar springs of low-alloy nickel-chromium-molybdenum steel, with particular reference to the procedure used to pre-stress and test them.

Details are given of test results which have been obtained, on regular run-of-work products, and comparisons are made of the fatigue behaviour of torsion bars

(1) neither shot-peened nor pre-stressed

(2) shot-peened but not pre-stressed

(3) pre-stressed but not shot-peened

(4) pre-stressed and afterwards shot-peened.

Softening of Nickel-Alloy Steels by Cold Working

P. CHOLLET and H. P. TARDIF: 'Softening of Steels by Cold Working.'

Nature, 1957, vol. 180, Oct. 12, pp. 754–5.

The authors briefly refer to earlier observations of softening occurring as a result of small amounts of cold work, and report varying effects produced in three types of steel:

A.I.S.I. Type 4340, containing carbon 0·38–0·43, nickel 1·65–2·00, chromium 0·70–0·90, molybdenum 0·20–0·30, per cent.

Hy-Tuf steel: carbon 0·25, silicon 1·50, manganese 1·30, nickel 1·80, molybdenum 0·40, per cent.

Chromium stainless steel: carbon 0·30, chromium 13, per cent.

The results now reported are preliminary in nature, and further investigation is being made of work-hardening and work-softening phenomena.

Sulphide-Corrosion Cracking of Steel Bolts

See abstract on p. 22.

Influence of Cadmium Plating on Properties of Ultra-High-Strength Steels

E. P. KLIER, B. B. MUVDI and G. SACHS: 'Design Properties of High-Strength Steels in the Presence of Stress Concentrations and Hydrogen Embrittlement. Part III. The Response of High-Strength Steels in the Range of 180,000–300,000 p.s.i. to Hydrogen Embrittlement from Cadmium Electroplating.'

Wright Air Development Center Tech. Report 56–395, *Pt.* III, Mar. 1957; 116 pp.

The report is introduced by a discussion on the theoretical aspect of hydrogen embrittlement caused by electrodeposited coatings and the initiation and propagation of cracking in high-strength steels. This

is followed by a highly detailed report on the influence of cadmium coatings on the fatigue strength of seven typical ultra-high-tensile steels of the following types:

A.I.S.I. 4340 (low-alloy nickel-chromium-molybdenum steel)

Vanadium-modified 4330 (low-alloy nickel-chromium-molybdenum steel)

98B40 (boron-treated lean triple-alloy steel)

Tricent (nickel-chromium-molybdenum-silicon-vanadium steel)

Crucible UHS 260 (chromium-manganese-silicon-molybdenum steel)

Hy-Tuf (nickel-chromium-manganese-silicon-molybdenum steel)

Super TM-2 (2 per cent. nickel-chromium-molybdenum steel)

The steels were heat-treated to tensile strengths varying from about 180,000 to 300,000 p.s.i. (80·5 to 133·5 tons per sq. in.). Susceptibility to embrittlement by cadmium plating was evaluated by sustained-load, fatigue and bend tests, and selected specimens were analysed, after plating, for hydrogen content.

The results revealed that—

(1) Hydrogen introduced into specimens electroplated with cadmium may be as high as that introduced through severe cathodic impregnation. Baking of the plated specimens sufficiently to restore ductility in bend test pieces led to no significant change in average hydrogen content, and it is therefore assumed that the change in properties results from a re-distribution of the hydrogen brought about by diffusion induced by baking.

(2) By suitable adjustment of the applied load, all the steels, at all the strength levels used, can be made to fail under sustained loads by cadmium plating.

(3) The susceptibility of a cadmium-plated steel to failure under sustained load depends on the severity of the notching conditions and on the metallurgical structure of the steels. The types studied fall into two groups, one comprising those which with rising tempering temperature become increasingly insensitive to the embrittling action of cadmium plating, and the second those which possess maximum insensitivity after tempering at intermediate temperatures. The first group is characterized by low silicon: the second group is silicon-rich.

(4) Although all the steels examined can be made to fail after cadmium plating, it is possible to secure good resistance to such embrittlement at strength levels as high as 270,000 to 280,000 p.s.i. (120·5 to 125 tons per sq. in.). Structural control is essential.

(5) The high-load low-cycle fatigue test is considered to hold promise as a means of determining notch-sensitivity of a steel. Bend tests, as used in this investigation, offer a simple and reasonably adequate means for evaluation of susceptibility to embrittlement by cadmium plating.

Automatic Direct-Reading Spectrograph

See abstract on p. 2.

Stress Relief in Weldments

See abstract on p. 2.

HEAT- AND CORROSION-RESISTING MATERIALS

Nickel-Chromium Alloys: Constitution and Structure

R. O. WILLIAMS: 'Nature of the Ni-Cr System.' *Trans. Amer. Inst. Mining and Metallurgical Engineers*, 1957, vol. 209, pp. 1257-60; *T.P.* 4621*E*; *Jnl. of Metals*, Oct. 1957. *Pt.* II.

Report of experiments made with the aim of determining the nature of ageing in both terminal solid solutions and the character of the phase diagram. Five alloys were examined, of which the compositions are given in the table below:

	Alloy No.				
	1	2	3	4	5
Nickel, wt. % ..	16·0	23·6	26·4	31·9	60
,, at. % ..	14·4	21·5	24·1	29·4	57

From the results the author draws the following conclusions:

'The solubility of nickel in chromium decreases very rapidly with decreasing temperatures. The solubility of chromium in nickel is in fair agreement with TAYLOR AND FLOYD' results (*Jnl. Inst. Metals*, 1951-52, vol. 80, pp. 577-87; *Nickel Bulletin*, 1952, vol. 25, Nos. 8-9, p. 202). The ageing in nickel-rich alloys at about 500°C. is not attributable to precipitation of chromium.

'The chromium-rich alloys are characterized by very rapid precipitation during cooling, which can be prevented only by very effective quenching. This rapid precipitation results from the rapidly decreasing solubility, the large heat release, and good atomic matching between phases.

'X-rays and microstructures show that there are several modes of precipitation (perhaps four). The (110) is the habit plane in the matrix and is parallel to the (111) of the precipitate. Diffuseness of X-ray lines indicates coherency and/or large strains in most cases. In some cases, close-packed-hexagonal lines have been found where the basal plane is identical with the (111) of the face-centred-cubic precipitate.

'The evidence showed that neither mechanical stress, low temperatures, nor both could produce transformations in these supersaturated chromium alloys.

The very high hardness and brittleness found in these alloys are the result of precipitation. No magnetic structures were found.

'No evidence has been found to support a recently proposed version of the Ni–Cr diagram. (*Trans. Amer. Inst. Mining and Metallurgical Engineers*, 1955, vol. 203, p. 127.) If a eutectoid (or peritectoid) reaction exists its temperature is above 1250°C.'

It is concluded that present knowledge of the nickel–chromium system is very incomplete.

Cast Iron-base Heat-Resisting Alloys

F. EBERLE, J. H. HOKE and W. E. LEYDA: 'Development of Cast Iron-base Alloys of Austenitic Type for High Heat-Resistance and Scale-Resistance.'

Wright Air Development Center Tech. Note 55-290, *Pt.* II, Jan. 1957; 86 pp.

The report presents the results of a comprehensive investigation carried out to meet the specification requirement for iron-base alloys suitable for use as substitutes for Haynes Stellite Alloy 21 (cobalt-base alloy containing carbon 0·20–0·35, chromium 25–30, molybdenum 4·5–6·5, nickel 1·5–3·5, per cent., with small amounts of other elements). It was laid down that the 'substitute' alloys must contain not less than 45 per cent. of iron and that they should

(*a*) have a fully austenitic structure,

(*b*) show resistance to oxidation at 2000°F. (1095°C.) equal to that of H.S.21,

(*c*) possess a creep-rupture strength at 1600° and 1800°F. (870° and 980°C.) equal to that of H.S.21,

(*d*) be suitable for fabrication by the investment-casting or shell-moulding process.

As a means of selecting the preferred basis compositions, tests were made on a number of promising alloys at the temperatures specified. Combinations, with iron, of chromium + nickel, chromium + aluminium + nickel, chromium + silicon + nickel, silicon + nickel and aluminium + nickel, were tried. Only iron-nickel-chromium compositions were found to offer a promising basis, since silicon and aluminium contents of more than 1 per cent. caused deterioration in rupture strength. The two most promising base compositions were found to be

Chromium 20, nickel 27–28, iron 45, per cent.

and

Chromium 15, nickel 32, iron 45, per cent.,

both alloys containing also carbon 0·50, manganese 1·0 and silicon 0·50, per cent.

Detailed study was then made of the possibility of enhancing the properties of these alloys by additions of niobium, tantalum, titanium, zirconium, molybdenum, tungsten, boron and nitrogen, in total concentrations of 4·0–7·5 and in varying combinations. These tests resulted in establishment of two outstanding alloys, one based on each of the parent compositions shown above. The compositions of the modified alloys are tabulated below:

Further tests were made with a view to developing still higher rupture strength, by varying the ratios of the strengthening elements and of the basis constituents. Evaluation of all the alloys was by rupture tests of up to 500 hours' duration at 1600° and/or 1800°F. (870° and/or 980°C.).

The second phase of the investigation, which is the subject of this report, covered further creep-rupture tests on the promising alloys, at 1600° and 1800°F. (870° and 980°C.), and selection of alloys for further study. The choice eventually centred on 'preferred alloy 9521' (see table), and various modifications of composition and treatment were made in an effort to secure properties even better than those characteristic of that alloy. Two of the modified alloys proved to be of particular interest:

(1) Alloy C 1186, containing carbon 0·5, silicon 0·5, manganese 1·0, chromium 20, nickel 27, niobium/tantalum 1·7, tungsten 3·3, cerium/lanthanum 0·25, per cent.

(2) Alloy 1228 (in which 12 per cent. was nickel replaced by 12 per cent. of cobalt.)

Study of various modifications of these alloys led to further tests on an alloy derived, by means of complex modifications, from the cobalt-modified alloy 1228. This material was of the following composition: carbon 0·5, silicon 0·5, manganese 1·0, chromium 15, nickel 20, cobalt 12, niobium/tantalum 2, molybdenum 2, tungsten 2, cerium/lanthanum 0·25, per cent.

The report contains extensive data on test results obtained with these basis materials modified by a variety of addition elements, in combinations too numerous and varied to be recorded in abstract. The criteria by which they were evaluated included creep-rupture, oxidation-resistance, resistance to thermal shock, cold ductility, and other properties relevant not only to service conditions but to fabrication.

As a result of the series of tests completed to date, the types of alloy shown in the table on p. 15, are under final consideration, as most nearly meeting the specification requirements.

	C %	Mn %	Si %	Cr %	Ni %	Nb/Ta %	Mo %	W %	CeLa %
Preferred Alloy 9521 ..	0·5	1	0·5	20	27	2·5	—	2·5	0·25
Alloy 954	0·5	1	0·5	15	32	2	2	2	0·25

Iron-base Casting Alloys Chosen for Further Study
(*see abstract on p. 16*)

Alloy	C %	Si %	Mn %	Cr %	Ni %	Co %	Nb/Ta %	Mo %	W %	Al %	Ce/La %
1388	0·5	0·5	1·0	15	20	12	2	2	2	—	0·25
1493	0·5	0·5	1·0	20	20	20	2	2	2	—	0·25
1228	0·5	0·5	1·0	20	15	12	2·5	—	2·5	—	0·25
C1186	0·5	0·5	1·0	20	27	—	1·7	—	3·3	—	0·25
9521	0·5	0·5	1·0	20	27	—	2·5	—	2·5	—	0·25
968	0·5	0·5	1·0	17	30	—	2·5	—	2·5	1	0·25

The all-over conclusion drawn is quoted below:

'Alloys containing not less than 45 per cent. iron, and having a minimum carbon content of 0·5 per cent., chromium not less than 20 per cent., and between 27 and 32 per cent. of nickel (part of which may be replaced by cobalt) can develop a creep-rupture strength at 1600°–1800°F. (870°–980°C.) approaching that of the cobalt-base alloy H.S.21 when they are strengthened by not more than 5 per cent. additions of niobium, tantalum, molybdenum and tungsten, in certain combinations and relative concentrations. These high-creep-rupture-strength iron-base alloys are equal to H.S.21 in oxidation-resistance at 2000°F. (1095°C.) but inferior to it in short-time cold and hot tensile strength, in cold ductility, and in resistance to thermal shock and thermal fatigue. The possibility of improving the latter by lowering the carbon content and compensating the resulting decrease in rupture strength with suitable modifications in composition has not yet been explored.'

Nickel Alloys for High-Temperature Service

R. P. DOMINIC: 'New Nickel Alloys for High Temperature Service.'

Materials in Design Engineering, 1957, vol. 46, Sept. 6, pp. 115–19.

The paper presents information on heat-treatment, properties and high-temperature applications of Inconel 700, 702, 713C, Incoloy T and Incoloy 901. The information given is taken from data sheets issued by The International Nickel Company, Inc., abstracts of which have appeared in the *Nickel Bulletin* (see appropriate items in the 1956 and 1957 volumes).

Elevated-Temperature Properties of Constructional Alloy Steels

See abstract on p. 10.

Notch Sensitivity at High Temperatures

H. R. VORHEES and J. W. FREEMAN: 'Notch Sensitivity of Heat-Resistant Alloys at Elevated Temperatures. Part 3. Final Data and Correlations.'

Wright Air Development Center Report 54-175, *Pt.* 3, Sept. 1956; 80 pp.

Increasing use is being made of rupture tests on notched specimens, in order to evaluate the behaviour of materials under sudden concentrated and complex stresses at elevated temperatures. To date, notch-rupture data have been used mainly as a means of comparing different alloys, and it has been shown that whereas the presence of a notch shortens the life of some materials, in others there is, within some ranges of temperature, a notch-strengthening effect (see, for example, papers abstracted in *Nickel Bulletin*, 1955, vol. 28, No. 8, pp. 132–3).

The investigation covered by the present report and its earlier parts was planned as a fundamental study of the mechanism responsible for this variation in notch response. Special attention was to be directed to the significance of stress re-distribution by a creep-relaxation effect. On the assumption that a notch, *per se*, should introduce no new properties in any material (but should merely modify stress-strain histories of fibres in the notched bar), the aim was to explain notch-rupture characteristics in terms of the properties of an alloy as normally measured in simple tension.

Part 1 of the report contained the results of preliminary tests on three turbine-blade alloys (S-816, Inconel X-550 and Waspaloy). The observations made indicated that notch-strengthening accompanied conditions of rapid relaxation and that notch-weakening was found when smooth-bar tests indicated slow relaxation even at stresses where rupture life was quite short.

Further work, reported in Part 2, confirmed that widely differing rates of stress re-distribution by creep relaxation could satisfactorily explain variations in notch behaviour. Notch strengthening was found to be associated with rapid relaxation of high initial stresses. It was postulated that in conditions where relaxation was slow a large portion of the total life is used up quickly at the high stress near the notch, resulting in early fracture at that position and lowering of the life of the specimen below that of a smooth bar.

Part 3 of the report covers further experimental work on S-816 and Inconel X-550 and on two other materials, an air-hardening chromium-silicon-molybdenum-vanadium steel (17-22-AS) and an age-hardened aluminium alloy. The tests were made on flat specimens, both smooth and notched

at the edges. (In the course of these tests, some further attention was given to the influence of method of preparation of the notches, and to factors other than creep and rupture properties which might be inter-related with notch effects.)

Comparative rupture properties for smooth and notched specimens, from the whole investigation, are correlated in the present report, and a summarized evaluation is made of (*a*) the relation of notched-bar to smooth-bar behaviour, (*b*) the factors influencing the rupture characteristics of notched specimens at elevated temperatures, and (*c*) the notch-effect characteristics of the various materials studied. Among the principal conclusions drawn are the following:

'The controlling factor affecting notch-rupture behaviour was the rate at which the stress concentration from the notch was reduced by yielding during loading and by subsequent time-dependent creep-relaxation, compared to the rate at which rupture life was used up.

'Relatively low yield strengths, allowing considerable plastic flow to reduce effective stresses at the base of notches, prolong rupture time. Relatively rapid creep at the high stresses initially existing at the base of notches promotes increased rupture time. Both factors, particularly low yield strength in relation to the rupture strength, combine to make S-816 practically insensitive to notches.

'Yielding and creep relaxation reduce the effective stresses at the base of notches, through interaction of the complex stresses. The extent of both processes in the three principal directions is distributed in proportion to the relative magnitude of the individual principal stresses.

'Some alloys lose considerable rupture life from small amounts of yielding during loading. This was the primary cause for notch sensitivity in Waspaloy at 1350°F. (732°C.) and probably was involved in the notch weakening of Inconel X-550 at the same temperature.

'The geometry of test specimens is important, in that it controls the initial stress patterns after loading for the rupture tests.

'A triaxial stress, from circumferential notches in round specimens, is less damaging than a biaxial stress, from notches in the edges of flat specimens.

'Sufficiently low yield strengths and rapid stress-relaxation by creep result in prolonged rupture times, in comparison with smooth specimens. Because reduction of effective stress by yielding or creep is dependent on the extent of triaxiality, it follows that rupture life increases with severity of stress concentration up to a maximum.

'Increasing yield strengths and creep-resistance reduce the stress concentration at which the maximum strengthening occurs. Materials of high yield strength and high creep-resistance in relation to the rupture strength can suffer loss in rupture life from all degrees of stress concentration.

'Residual stresses from notch preparation can apparently influence performance of specimens which are resistant to stress relaxation.'

Brittle-to-Ductile Fracture in Chromium-base Alloys

See abstract on p. 8.

Compressive Stress-Strain Properties of Precipitation-Hardening Steels at Elevated Temperatures

B. A. STEIN: 'Compressive Stress-Strain Properties of 17-7 P.H. and A.M. 350 Stainless-Steel Sheet at Elevated Temperatures.'

Nat. Advisory Committee for Aeronautics Tech. Note 4074, Aug. 1957; 21 pp.

Precipitation-hardenable stainless steels, on account of their formability in the annealed condition and their capacity for thermal strengthening after fabrication, are of considerable interest to the aircraft designer. Compressive stress-strain data on such materials are, however, sparse, and the investigation described in this report was carried out to supply further information on this aspect. Compressive stress-strain tests were made, over the range room temperature to 1200°F. (650°C.), on sheet material of two typical precipitation-hardenable steels, of which the compositions are shown in Table I p. 17.

The heat-treatments applied and the resulting conditions in which the steels were tested, are shown in Table II, p. 17.

The compression-testing equipment is described and illustrated. The tests were made both in the direction of and across the rolling direction, and all specimens were held at test temperature for ½ hour before loading.

Representative stress-strain curves are given for both materials at the test temperatures. From these curves significant design data were obtained, such as compressive yield stress, Young's modulus, and secant and tangent moduli. These are presented in graphical and tabular form.

An empirical equation which describes the stress-strain curves is presented and the variation of the parameters in this equation with temperature is given for the temperature range investigated.

It was found that the thickness of the sheet and the direction in which the material was loaded produced no significant variation in the compressive mechanical properties. In the precipitation-hardened condition the yield strength of 17-7 P.H. steel fell rapidly at temperatures above 700°F. (370°C.): a parallel effect occurred in A.M. 350 above 800°F. (425°C.). For temperatures up to about 825°F. (440°C.) the compressive yield strength of 17-7 P.H. was higher than that of A.M. 350, but above that temperature the position was reversed.

Tests on the two materials in the annealed condition showed only slight loss of strength up to 1200°F. (650°C.), at which temperature the yield value for the annealed sheet was slightly higher than that found for the respective steels in the precipitation-hardened condition.

Table I

Precipitation-Hardening Stainless-Steels: Compositions and Heat Treatments

(*see abstract on p. 16*)

	C %	Si %	Mn %	S %	P %	Cr %	Ni %	Al %	Mo %	Fe %
17-7 P.H. ..	0·09 max.	1·00 max.	1·00 max.	0·03 max.	0·04 max.	16-18	6·5–7·75	0·75-1·50	—	Rem.
A.M.350 ..	0·08-0·12	0·5 max.	0·50-1·25	0·03 max.	0·04 max.	16-17	4-5	—	2·5-3·55	Rem.

Table II

Material	Nominal Sheet Thickness, in.	Condition as received	Heat-Treatment	Designation of condition after Heat-Treatment
17-7 P.H.	0·050	Annealed	Heated at 1400° ±25°F. for 1½ hr. Air cooled to 60°F. within 1 hr. after removal from furnace Heated at 1050° ±10°F. for 1½ hr. Air cooled	TH 1050
A.M.350	0·045 and 0·063	Annealed	Heated at 1350° ±25°F. for 1½ hr. Air cooled Heated at 850° ±25°F. for 1½ hr. Air cooled	Double-aged

Mechanical Properties of Structural Materials after Rapid Heating

J. D. MORRISON and J. R. KATTUS: 'Tensile Properties of Aircraft Structural Metals at Various Rates of Loading after Rapid Heating.'

Wright Air Development Center Tech. Report 55-199, *Pt.* II, Nov. 1956; 181 pp.

This report is a further contribution to the literature relating to the load-carrying properties of materials as affected by rapid heating and by exposure for relatively short periods to high temperatures.

The first part of the report had included a literature survey, the development of suitable test equipment, and experimental determinations of the properties of seven aircraft structural materials in sheet form: titanium-stabilized stainless steel, two titanium alloys, a magnesium alloy and three Alclad aluminium alloys. The results were reported in *W.A.D.C. Technical Report* 55-199, *Pt.* I. A brief résumé of the test conditions used is given in the present report; for fuller details attention is directed to Part I.

The second series of tests included determination of the properties of the following materials:

Sheet Materials

Stellite 25 (cobalt-base alloy containing nickel, chromium and tungsten).

Sheet Materials (cont'd)

Inconel X (precipitation-hardening nickel-chromium-base alloy).

18-8 chromium-nickel steel (Type 301), half hard and full hard.

17-7 P.H. stainless steel, annealed and precipitation-hardened.

Chromium steel, normalized, and quenched-and-tempered.

Carbon steel, hot-rolled.

Titanium alloys.

Cast Materials

Magnesium alloy.

Aluminium alloy.

The purpose of the work was to determine the effects of variations in strain rate and holding time, on the tensile properties of these materials after they had been heated within 10 seconds to test temperatures up to 1200°F. (650°C.). Major attention was directed towards the influence of such treatment yield and ultimate strengths: other properties, such as modulus of elasticity, limit of proportionality and total elongation, were also determined, but only for the purpose of establishing general trends. The investigation covered strain rates varying from 0·00005 in./in./sec. to 1·0 in./in./sec. and holding times at test temperature from 10 seconds to 30 minutes.

With few exceptions, the strength properties of the materials examined decreased with rising temperature over the range 85°–1200°F. (30°–650°C.). The exceptions were the A.I.S.I. 4130 chromium steel and the carbon steel, over limited ranges of temperature and rates of strain. In these materials increase in strength occurring with rising temperature was probably caused by blue brittleness.

Modulus of elasticity tended to fall with rising temperature, but between 400° and 800°F. (205° and 425°C.), at certain rates of strain, an increase in modulus was found in several of the materials. Percentage elongation varied erratically with rising temperatures.

In general, tensile strength increased as the strain rate was increased from 0·00005 in./in./sec. to 1·0 in./in./sec. Percentage elongation varied inconsistently with variations in strain rate, depending upon the material, the temperature and the time of holding. Modulus-of-elasticity values were not greatly affected by changes in strain rate or holding time.

Up to 1000°F. (540°C.) 17-7 P.H. stainless steel in the precipitation-hardened condition (T.H.1050) had the highest strength of any of the materials examined: in the range 1000°–1200°F. (540°–650°C.) precipitation-hardened Inconel X was superior to any other.

The results indicate a rough qualitative relationship between tensile properties and creep characteristics. The tensile properties of materials which have good creep strength are apparently less sensitive to changes in rate of strain than are those which show poor creep properties.

Rapid-Heating Tests on Inconel X

I. M. KURG: 'Tensile Properties of Inconel X Sheet under Rapid-Heating and Constant-Temperature Conditions.'

Nat. Advisory Committee for Aeronautics Tech. Note 4065, Aug. 1957; 20 pp.

The development of ultra-high-speed aircraft and missiles has confronted the designer with the problem of aerodynamic heating. Tests at the elevated temperatures involved have shown that materials considered suitable for such applications exhibit higher tensile strength at elevated temperatures when rapidly heated than when tested under conventional constant-temperature conditions. Investigations of the behaviour of sheet materials rapidly heated have included tests on various aluminium alloys, Inconel, titanium alloy and magnesium alloys: the results for Inconel sheet were published in *N.A.C.A. Technical Note* 7731 (see *Nickel Bulletin*, 1957, vol. 30, No. 2, p. 42). Later related work, on Inconel X, is described in the present report.

Test specimens were in the form of 0·05-in. thick sheet, heat-treated for one hour at 1400°F. (760°C.) and air-cooled. The composition of the alloy used is given as nickel 72·77, chromium 14·98, iron 6·82, titanium 2·45, niobium + tantalum 0·96, aluminium 0·76, silicon 0·37, manganese 0·74, copper 0·06, carbon 0·06, sulphur 0·06, per cent.

Two series of tests were made:

(1) Sheet was heated to failure at nominal temperature rates of 0·2°–100°F. per second, under constant tensile-load conditions.

(2) For comparison, conventional tensile stress/strain values were obtained under elevated-temperature conditions.

The results are similar in nature to those established for Inconel. At each stress level the temperatures of both yield and rupture rose with increase in rate of heating. Comparison of the results with data obtained from conventional tensile tests showed that for rates of heating of 0·2°F. per second and less, the yield and ultimate-stress values in rapid-heating tests were lower than those obtained in constant-temperature conditions, but that with heating rates of 2°F. upwards they were appreciably higher.

Correlation of the results made possible the construction of master curves from which stresses and temperatures for yield and rupture can be predicted under rapid-heating conditions.

British and U.S. Steel Specifications

See abstract on p. 9.

Production of Holes in Refractory Alloys by Spark Erosion

'Spark Erosion of Deep Holes on a Wickman Erodomatic Machine.'

Machinery (*Lond.*), 1957, vol. 91, Oct. 11, pp. 867–70.

Recently developed techniques for the manufacture of cooled blades for gas turbines involve machining of deep holes of small diameter in the nickel-base and other alloys used (e.g., holes 0·030-in. in diameter and 4-in. deep). Due to the work-hardening properties of these refractory alloys, formation of such holes by normal drilling methods is difficult and research has been carried out to determine the feasibility of 'drilling' by spark erosion.

This article describes spark-erosion procedure developed by Wickman Ltd., of Coventry. The technique is illustrated, *inter alia*, by reference to holing of Nimonic 90 by spark erosion, using the Wickman Erodomatic machine. The writer discusses the advantages of the main features of the method, and the nature of the machine and the ancillary equipment used.

Nickel-containing Alloys in Aircraft Gas Turbines

The technical press has recently published detailed and well-illustrated descriptions of a number of power units used in some important British aircraft. Notes on the scope of the respective articles are given below. In all cases reference is made to the extensive use of high-nickel alloys in the engines described, e.g., alloys of the Nimonic series for rotor blading, flame tubes and other components.

'The Versatile Orpheus.'

Aeroplane, 1957, vol. 93, Nov. 22, pp. 779–82.

Review of the evolution of the Orpheus lightweight turbojet, from the inception of the engine as the power unit for the Folland Gnat (in which the initial flight experience of the engine was gained), through large-scale development by the Bristol Aeroplane Company, to its present version, the B Or.3, which has a design rating of 4,850-lb. thrust. Within three years of its first run the engine has been adopted as the power plant for fourteen new types of aircraft. The most recent version, the B Or.12, is intended to have a thrust of 8,000 lbs. The article includes a cut-away drawing of the engine, with details of the individual components.

See also

'Bristol Orpheus: 'The Heart of the Gnat.'

Flight, 1957, vol. 72, Nov. 22, pp. 809–12.

Historical survey of the engine's development, with comments on its use in various aircraft. An interesting feature of the article is the photographs of some of the main engine components: these include the rotating assembly of compressor, shaft and turbine; the single-stage turbine (the rotor-blades of which are of Nimonic 90) and the compressor delivery casing in R.R.250 alloy.

Both the above articles are complementary to others in the same journals, which give details of the Folland Gnat Mk. I aircraft.

'Double Mamba A.S.M.D.8. Development of Armstrong Siddeley's Double Turboprop.'

Flight, 1957, vol. 72, Nov. 22, pp. 815–19.

The development of the Mamba-type engines is traced, from their genesis in the single Mamba of 1945 through to the Double Mamba A.S.M.D.8 (comprising two engines coupled to drive co-axial propeller shafts but otherwise operating independently). This unit (rating 3600 s.h.p.) has doubled the power which would have been available in a double engine of equivalent design based on the earliest single engine. Detailed cut-away drawings serve to illustrate the comprehensive information presented in the text. It is of interest to note that in the new engine advantage has been taken of the properties of Nimonic 100, which is used for the first- and second-stage rotor blades.

'Fairey's Vertical Take-off Transport.'

Aeroplane, 1957, vol. 93, No. 15, pp. 700–3, 753–4; drawing, pp. 726–7.

The article contains a comprehensive description of the prototype Fairey-Rotodyne helicopter, which, in addition to a single four-bladed rotor, has the wings and tail unit of a normal aeroplane. It thus combines the advantages of vertical take-off with the optimum efficiency in flight conferred by two Napier Eland turboprop engines mounted in the wings. The detailed information given on the structural components of the aircraft are supplemented by notes on materials employed in the Napier Eland N.El.3 turboprop units used in the prototype. The production aircraft are to be equipped with the 3,500-e.h.p. Eland NEl.7, and an indication of the increased power, and, associated higher gas temperatures, which will be obtained in this unit is the substitution, in the N.El.7, of Nimonic 100 and Nimonic 90 for the Nimonic 90 and Nimonic 80A alloys used, respectively, in the first-stage and the second- and third-stage rotor blades of the earlier engine. The tip-pressure jets of the rotor blades are made entirely of Nimonic alloys.

Oxidation Characteristics of a Copper-Nickel Alloy

J. A. SARTELL, S. BENDEL, T. L. JOHNSTON and C. H. LI: 'The Effect of Oxide Recrystallization on the Oxidation Kinetics of a 62 : 38 Copper-Nickel Alloy.'

Trans. Amer. Soc. Metals, 1957, vol. 50, *Preprint* 47; 24 pp.

Report of study of the oxidation behaviour of the alloy at temperatures in the range 760°–927°C.

It was found that during isothermal oxidation two oxide layers are formed: the outer one, consisting of CuO, the growth of which is described by a single parabolic law, and an inner one, consisting of a mixture of Cu_2O and NiO, of which the growth is described by two parabolic laws. The growth of the CuO layer requires the outward movement of cupric ions, whereas the oxide front of the inner layer moves forward by nucleation and growth of NiO, resulting from an internal-oxidation mechanism which involves rapid inward diffusion of oxygen from the $CuO:Cu_2O$ interface to the Ni-Cu solid solution. Change in the parabolic rate constant associated with the growth of the inner $NiO-Cu_2O$ layer is caused by recrystallization of the outer CuO layer.

Nickel-containing Aluminium Alloys for Contact with High-Temperature Water

J. E. DRALEY and W. E. RUTHER: 'Experiments in Corrosion Mechanism: Aluminium at High Temperatures.'

U.S. Atomic Energy Commission Report ANL-5658, Apr. 1957; 26 pp.

Further to earlier reports on the corrosion of aluminium-base alloys in water at elevated temperatures, and the inhibition of attack by means of a protective phase formed in alloys containing small amounts of nickel and other elements, the present report continues to develop the hydrogen hypothesis which had been advanced to explain the 'blister' corrosion observed. (For reference to earlier literature, see abstracts in *Nickel Bulletin*, 1956, vol. 29, No. 7, pp. 145 and 142; 1957, vol. 30, No. 12, p. 231.)

Experiments in which high-purity aluminium and aluminium alloys containing 0·5 and 1 per cent. of nickel were exposed to distilled water at temperatures of 290°–350°C. and other tests, in superheated steam, confirmed interpretation of the corrosion mechanism in terms of gas pockets or blisters.

Further support was derived from results of subsequent tests. The resistance to disintegrating attack conferred by the cathodic second phase formed by suitable alloying was shown to increase with the amount of the phase present and with progressively fine dispersion.

Further points of interest which emerged from the tests reported were as follows: (1) attempts to reduce still further the rate of corrosion of resistant alloys by alloying the aluminium matrix with a solid-solution additive were mainly unsuccessful; (2) experiments with aqueous corrosion inhibitors indicated that at pH 3·5 phosphoric acid effects a considerable reduction in the equilibrium corrosion rate of the nickel-modified alloy at 290°C., but further work is required to assess the practical value of this and other inhibiting media.

Tests in superheated steam demonstrated that cathodic second phases confer corrosion-resistance in that medium, as in water. An aluminium alloy containing 5·5 per cent. of nickel, exposed for two weeks in steam (400°C.; 4,000 p.s.i.g.) showed resistance to corrosion superior to that of Zircaloy.

Nickel-Zirconium System

See abstract on p. 6.

Influence of Boron on Structure of Nickel-Chromium Steels

C. C. CLARK, J. R. MIHALISIN and K. G. CARROLL: 'Investigation of Grain-Boundary Phase in Stainless Steel.'

Trans. Amer. Inst. Mining and Metallurgical Engineers, 1957, vol. 209, pp. 1210–11; *T.N.* 401*E*.

Jnl. of Metals, Oct. 1957.

The note describes investigation of an unusual grain-boundary precipitate (found to be a carbide) formed in a 25-20 chromium-nickel steel on cooling in air from 2000°F. (1093°C.). A specimen water-quenched from the same temperature showed no sign of this phase.

Chemical analysis of the steel indicated a boron concentration of about 0·001 per cent. by weight, and examination of other heats of a similar steel demonstrated that the quantity of network phase present after air cooling was sensitive to the amount of boron present. Since the network forms during the short time in which the steel cools through the temperature range of carbide formation, it is suggested that the boron present powerfully catalyses that reaction.

Boron-containing Stainless Steel for Use in Atomic Energy Plant

J. A. BERGER and W. L. KEENE: 'A New Stainless for Atomic Energy.'

Steel, 1957, vol. 141, Sept. 9, pp. 104–7.

Boron has proved to be a valuable addition to corrosion-resisting nickel-chromium steels used in atomic-energy applications. An 18-8 chromium-nickel steel containing 1 per cent. of boron is being used as a thermal shield in reactors and a 2 per cent. boron steel is employed for control rods. This article describes methods which have been developed to overcome the difficulties resulting from the inherent hot-shortness of steels containing boron in percentages of the order required for such purposes. It also refers to clad materials, in which the benefits resulting from boron content are combined with high corrosion-resistance, by means of a boron-containing steel for the core, clad with normal austenitic stainless steel.

Effect of Irradiation on Corrosion of Stainless Steel in Uranyl-Sulphate Solutions

F. H. SWEETON, M. D. SILVERMAN and H. O. DAY: 'The Effect of Irradiation on the Corrosion of Type 347 Stainless Steel by Uranyl-Sulphate Solutions.'

U.S. Atomic Energy Commission Report O.R.N.L.-2284, 1957; 25 pp.

The authors open with a résumé of the unusual combination of properties necessary in materials which can be considered for use in aqueous homogeneous reactors. The extensive store of information which has been built up on the properties of niobium-stabilized stainless steel (chromium 17–19, nickel 9–13, per cent.) indicated that such a material would function satisfactorily in the conditions involved, and it was accordingly chosen for use in the first aqueous homogeneous reactor of the circulating-fuel type built in the U.S.A. This report summarizes the considerable amount of information which was assembled with regard to the corrosion-resistance of the steel during the period 1952–1954. The data presented are based on experiments both in and out of pile: information is given also on the techniques which were used to evaluate the behaviour of the steel.

The observations made appear to indicate that reactor radiation above a power-density level of 4 Kw./ml. causes a sharp rise in rate of corrosion, but it appears probable that the mechanism is not a simple function of radiation levels or of the concentration of fission products. Pre-treatment by exposure of the steel to the test solution without irradiation inhibited corrosion for a period which appeared to depend on the rate of deterioration of the protective film formed out of pile. The influence of temperature, in the range investigated (250°–290°C.), was found to be insignificant. Potassium pertechnetate proved ineffective as a corrosion inhibitor.

Influence of Irradiation on the Properties of Metals

See abstract on p. 1.

Ductility of Stainless Steel: Possible Influence of Carbide Precipitation

A. KRAME and W. M. BALDWIN: 'Carbide Precipitation and Brittleness in Austenitic Stainless Steel.'

Trans. Amer. Soc. Metals, 1957, vol. 50, *Preprint* 32; 18 pp.

It has been recognized for many years that austenitic

steels lose ductility when deformed at high rates of strain and it has been generally assumed that strain-rate embrittlement is related to the martensite transformation which occurs during plastic deformation. This explanation has, however, recently been challenged by the results of investigations by FORM and BALDWIN (*ibid.*, 1956, vol. 48, pp. 474-85: *Nickel Bulletin*, 1956, vol. 29, Nos. 1-2, p. 20). They concluded that embrittlement must be explained on some basis other than the $\gamma \rightarrow \alpha$ transformation. On the presumption that carbide precipitation* could be a significant factor, a search was made of the literature, but with little positive result. The work reported in the present paper was therefore carried out to throw further light on this point.

To evaluate the relationship between ductility, rate of strain, temperature of deformation and carbide precipitation, tensile tests were conducted, at various rates of strain (0·05 to 19,000 in./in./min.), at temperatures ranging from −321° to + 500°F. (−196° to +260°C.), on steels of varying carbon content. The steels were tested in the annealed and the sensitized conditions. Magnetic measurements on broken specimens, and oscillograph readings, served to detect and assess the amount of martensite formed during the tests.

The steels used were of the compositions shown in the table below.

The following conclusions are drawn from the results of the various tests:

For the *annealed material* the findings are in complete agreement with those reported by Form and Baldwin:

'(1) At low strain rates, as the test temperature was varied from −321°F. to +500°F. (−196° to +260°C.); a maximum in tensile ductility occurred at room temperature.

'(2) At high strain rates the steels gained steadily in ductility as the test temperature was raised.

'(3) At constant temperature, stainless steels lost ductility as the rate of strain increased; the greatest loss occurred at room temperature.

'(4) The amount of martensite formed during deformation cannot be correlated with the ductility.

* The authors note that the effect of carbide precipitation on intergranular failure under corrosive conditions is, of course, well known. The present paper is concerned with purely mechanical effects.

For the *sensitized materials:*

'(5) The degree of sensitization was a function of the carbon level. Type 304 ELC (0·024% C.) was not embrittled, whereas Type 304 (0·060% C.) was embrittled, but to a lesser extent than Type 302 (0·090% C.).

'(6) Carbide embrittlement manifested itself primarily in the range of low temperatures at low rates of strain.

'(7) The carbide-embrittled specimens fractured intergranularly.'

It is thus obvious that carbide precipitation does not explain the high-speed embrittlement encountered in austenitic stainless steels as a class, and magnetic measurements of martensite formed during deformation confirmed earlier results that the $\gamma \rightarrow \alpha$ transformation is also incapable of giving a full and adequate explanation of the phenomenon.

Corrosion of Austenitic Stainless Steels in Sulphuric Acid

E. H. PHELPS and D. C. VREELAND: 'Corrosion of Austenitic Stainless Steels in Sulphuric Acid.' *Corrosion*, 1957, vol. 13, Oct., pp. 619t-24t.

In view of the wide use of chromium-nickel stainless steels for chemical plant, and of the many processes in which sulphuric acid is employed, considerable research has been carried out on the resistance of stainless steels to corrosion by this medium. The authors briefly refer to some of the outstanding literature, and describe experiments designed to establish the behaviour of austenitic stainless steels in air-saturated and air-free sulphuric acid, in concentrations of 0·1 to 96·0 per cent. by weight, at temperatures of 30° and 50°C. Methods used for producing the two conditions in the solutions are described and a full account is given of the test technique. In addition, preliminary results are presented of a laboratory study of the use of potential measurements as a means of estimating the susceptibility of stainless steels to corrosion.

The tests were made on six types of austenitic stainless steel (representative of commercially available materials), solution-annealed at 1950°F. (1065°C.): straight 18-8 and 25-20 chromium-nickel types, 'high 18-8' containing varying amounts of molybdenum, and titanium- and niobium-stabilized '18-8'. The test periods were usually 96 hours,

Steels used in Carbide-Precipitation Tests

Type	C %	Si %	Mn %	S %	P %	Ni %	Cr %	Mo %	Cu %
304 ELC	0·024	0·49	1·33	0·018	0·024	10·12	19·00	0·09	0·14
304	0·060	0·55	1·38	0·030	0·029	9·47	18·49	0·20	0·21
302	0·09	0·69	0·88	0·010	0·040	9·27	18·70	0·24	0·13

except in the case of specimens which corroded very rapidly. Corrosion rates were calculated on the basis of loss of weight.

The results of the tests show that the steels remain passive until a critical acid concentration is reached, at which the rate of attack increases with increase in acid concentration. At very high concentrations the corrosion rate tends to decrease. The critical concentration appeared to depend on alloy content: *inter alia*, it was shown that the more highly alloyed Type 310 was more susceptible to the effects of oxygen in the acid solutions than was an '18-8' type. It is concluded that the agreement between the potential measurements and the passive-active regions determined by the more direct loss-of-weight method is sufficiently good to warrant further investigation of the usefulness of the electrochemical test under operational conditions.

Sulphide-Corrosion Cracking of Bolts

D. WARREN and G. W. BECKMAN: 'Sulphide-Corrosion Cracking of High-Strength Bolting Material.'
Corrosion, 1957, vol. 13, Oct., pp. 631t–46t.

The work reported in this paper originated as a result of frequent incidence of sulphide-corrosion cracking in bolts of low-alloy chromium-manganese-molybdenum steel. The aim of the investigation was to obtain a clearer insight into the mechanism of failure and, in particular, to secure data permitting modification of design which might diminish the susceptibility of the steel to such deterioration.

Bolts were used as test specimens because their stress patterns are difficult to simulate with any other type of laboratory specimen. In order to assess the possibility of improvement by means of material, as well as design, bolt specimens of other materials were also tested: austenitic stainless steels, K Monel, Inconel, Inconel X, aluminium bronze and an aluminium alloy. The effects of protective metallic coatings, and of adding inhibitors to the corroding solutions, were also studied as a means of reducing corrosion on the low-alloy-steel bolts.

The tests were made in three environments of aqueous hydrogen sulphide: (1) at 40°C. and at a pressure of 250 p.s.i., the bolts being exposed in the vapour phase saturated with water; (2) at 120°C. and a pressure of 250 p.s.i., with the bolts in either the liquid or vapour phase, or a combination of both, and (3) at room temperature and atmospheric pressure. In most tests the specimens were exposed for a minimum of 3,000 hours.

Comprehensive data are reported on the cracking observed under each of the test conditions and as a function of all the variables investigated. The conclusions drawn are summarized as follows:

Susceptibility to cracking increased with increasing bolt hardness, increasing applied stress, increasing amounts of plastic deformation and decreasing test temperature.

(*a*) In the absence of plastic deformation, no bolt failures occurred at hardnesses below R_c 27, even under stresses approaching the yield strength of the material. In the hardness range of R_c 27–55 the cracking susceptibility increased with increasing hardness and increasing applied stress. For each hardness level, there appeared to be a minimum or threshold stress required to produce cracking. Increasing the hardness lowered the minimum stress for cracking.

(*b*) Plastic pre-straining of the bolts (1) lowered the minimum hardness for sulphide-corrosion cracking from R_c 27 to R_c 22, and (2) increased the number of bolt failures in the hardness range of R_c 27–33.

(*c*) Increasing the test temperature from 40°C. to 120°C. greatly reduced the tendency for sulphide-corrosion cracking. The pressure of the test system, in the range of 1–17 atmospheres, had little effect on occurrence of cracking.

(*d*) The time for failure by sulphide-corrosion cracking decreased sharply with increasing hardness, and decreased to a much lesser degree with increasing applied stress and increasing amounts of plastic deformation.

No connexion was established between the incidence of cracking and the corrosion rates in the three environments. The results indicated that in a uniformly martensitic structure both intergranular and transgranular cracking can occur.

In discussing the cause and mechanism of failure, the authors review the various theories which have been advanced and conclude that hydrogen embrittlement, rather than stress corrosion, is the main contributory factor. In support of this conclusion they refer to the decrease in susceptibility to cracking associated with rise in temperature and lowering of hardness level, both of which effects are features characteristic of hydrogen embrittlement. Further, when the belts were pickled under stress in an acid solution conducive to hydrogen embrittlement their behaviour under test was analogous to that observed in the hydrogen-sulphide environment.

Most of the coatings were to some extent effective in increasing bolt life, but they did not provide consistent or reliable protection against cracking. A relatively heavy nickel deposit (0·005 in. thick) gave good protection in the one environment in which it was tested. Addition of inhibitors reduced, but did not eliminate, failure. Of the alternative materials tested, nickel-chromium stainless steel, K Monel, Inconel and Inconel X proved completely resistant to cracking and showed a good level of general corrosion-resistance. Aluminium and the 5 per cent. aluminium bronze were the only 'substitute' materials which showed any susceptibility to cracking.

Pitting Corrosion in High-Alloy Materials

M. STERN: 'The Relation between Pitting Corrosion and the Ferrous-Ferric Oxidation-Reduction Kinetics on Passive Surfaces.'
Jnl. Electrochemical Soc., 1957, vol. 104, Oct., pp. 600–6.

Pitting of metals may be divided into two separate processes, pit initiation and pit propagation. The author refers to major literature on both aspects.

Discussion in the present paper is limited to the electrochemical factors involved in the pit-propagation process. Since it has been shown that the pitting process is essentially under cathodic control, the overvoltage behaviour for ferric-ion reduction on the cathode surface should influence the rate of growth of the pits, and in this work ferric-ferrous activation-overvoltage behaviour was studied on a series of alloys, in order to evaluate the significance of this aspect of the corrosion process in determining the varying pitting resistance of the respective materials. The materials used were low-carbon 18-9 chromium-nickel, 17-13-2·5 chromium-nickel-molybdenum and 25-20 chromium - nickel steels, Hastelloy F (nickel - chromium - iron - molybdenum alloy), Hastelloy C (nickel-chromium-molybdenum-iron - tungsten alloy), and titanium.

Intergranular Corrosion in High-Chromium Steels: Influence of Nickel Additions

J. R. UPP, F. H. BECK and M. G. FONTANA: 'Influence of Nickel on Intergranular Corrosion of 18 per cent. Chromium Steels.'

Trans. Amer. Soc. Metals, 1957, vol. 50, *Preprint* 51; 18 pp.

Study of the mechanism of intergranular corrosion in stainless steels and of methods for inhibiting it has revealed, *inter alia*, that a thermal treatment which renders the 18-8 austenitic chromium-nickel steels susceptible to such corrosion has a preventive effect on straight 16-18 per cent. chromium steels, i.e., what is a 'bad' treatment for the austenitic steels is 'good' for the ferritic type. The work reported in this paper was carried out to determine the nickel content at which the changed response to thermal treatment occurs, and to develop optimum treatments for steels of alloy content intermediate between the 16-18 per cent. chromium and the 18-8 chromium-nickel types.

The tests were made on twelve experimental heats of steel varying from zero up to 8·3 per cent. nickel, and on two commercial types representative, respectively, of 18 per cent. chromium steel and 18-8 chromium-nickel Type 304. A large amount of experimental data is given on the intergranular corrosion-resistance (standard boiling-nitric-acid test) of the steels in conditions resulting from various thermal treatments.

The results show that the change in mechanism of the intergranular corrosion takes place at about 2·5-3 per cent. of nickel. The recommendation is made that 18 per cent. chromium steels containing less than 2·5 per cent. of nickel should, from the point of view of thermal treatment, be considered as ferritic steels, while for those containing more than 3 per cent. of nickel treatments suitable for austenitic steels should be used. It was observed that furnace cooling from 2000°F. (1095°C.) of steels in the transition range of composition (2·0-3·5 per cent. of nickel) caused high susceptibility to intergranular attack. When water-quenched from 1400°F. (760°C.) none of the steels showed such corrosion.

Resistance of Metallic Materials to Cavitation-Erosion

AMER. SOC. MECHANICAL ENGINEERS: 'Resistance of Various Materials to Cavitation Damage.'
Report of 1956 Cavitation Symposium.
Published by the Society, 1957; 27 pp.

A feature of the Annual Meeting of the American Society of Mechanical Engineers was a Cavitation Symposium at which, by means of written reports and discussion, a large amount of information and opinion was brought together. All the material so made available has been edited for incorporation in this publication.

The following information, given in the foreword, demonstrates the scope of the report:

> 'The report is a condensation of 35 years of field and laboratory experience with the resistance of materials to cavitation damage. It outlines the experience operating companies from the United States and Canada have had with such materials as cast iron, cast steel, bronze, various types of chrome-nickel stainless-steel welds, cast-chromium steel, both cast and welded aluminium bronzes, rubber, and sprayed-on materials.
>
> 'The report details the practices of a number of operating companies with reference to frequency of inspection for cavitation damage, preparation of the metal surface for repair, repair procedure, and surface finish after repair.
>
> 'Upon comparing the results of laboratory tests made with various types of accelerated cavitation machines, it was found that the order of relative resistance of various materials to cavitation damage is practically the same, regardless of the type of machine used. It was also found that there is a close correlation between field experience and laboratory tests, showing that laboratory testing gives a good indication of what can be expected under field conditions.
>
> 'The resistance of new materials, such as manganese stainless steel, and new methods of application such as 'fuse-metallizing' are discussed.'

The final section of the report lists the order of resistance of various materials to cavitation, based on both field experience and laboratory tests. This list, shown below, indicates that stainless steels, certain types of bronze, and cast chromium stainless steels stand high in order of resistance.

Order of Cavitation-Resistance of Metals

Based on Field Experience:

1. Stellite
2. 17-7 Cr-Ni stainless-steel weld
3. 18-8 Cr-Ni stainless-steel weld
4. Ampco No. 10 weld
5. 25-20 Cr-Ni Weld
6. Eutectic-Xyron 2-24 weld
7. Ampco bronze castings
8. 18-8 Cr-Ni cast stainless steel
9. Nickel-aluminium bronze, cast
10. 13 per cent. Cr steel, cast
11. Manganese bronze, cast

12. 18-8 stainless spray-metallizing
13. Cast steel
14. Bronze
15. Rubber
16. Cast Iron
17. Aluminium

Based upon laboratory tests:

1. Stellite
2. Two layers 17-7 Cr-Ni stainless-steel weld
3. 18-8 Cr-Ni stainless-steel weld
4. Ampco No. 10 weld
5. Cast Ampco No. 18 bronze
6. Nickel-aluminium bronze
7. 18-8 Cr-Ni, cast stainless steel
8. 13 per cent. Cr, cast stainless steel
9. Manganese bronze, cast
10. Cast steel
11. Bronze
12. Cast iron
13. Sprayed stainless 18-8 Cr-Ni steel
14. Rubber
15. Aluminium

From the information now available, it is concluded that the next important step in the study of cavitation phenomena should be towards determining the relationship between velocity and cavitation damage. The establishment of such a relationship for various materials could greatly influence design and application of hydraulic structures and machines, and the materials used in their construction.

Mechanical Finishes for Stainless Steel

R. PARET: 'Selecting Mechanical Finishes for Stainless Steel.'
Materials in Design Engineering, 1957, vol. 46, Sept., pp. 111–14.

The article reviews the wide range of standard metal finishes obtainable on stainless-steel sheet, and the mechanical techniques used to produce them. The information given in the text is supplemented by tables showing the respective initial surfaces, the equipment and procedure required to produce the final surfaces, and typical applications of stainless steels having the various finishes. A brief résumé is made of barrel-finishing methods used to brighten small parts and components which are difficult to buff, and reference is made to textured patterns producible in stainless-steel sheet by passing the metal between engraved rolls.

Applications of Nickel-base Corrosion-Resistant Alloys

E. D. WEISERT: 'High Alloys to Combat Corrosion.'
Corrosion, 1957, vol. 13, Oct., pp. 659t–71t.

The author briefly notes the considerations which must govern the selection of high-alloy materials for corrosion-resisting applications. The economic aspect (initial price, maintenance costs, special techniques involved in fabrication and installation, etc.) must be balanced against the inherent corrosion-resistance of the materials, the extended life of plant obtained by their use, avoidance of breakdowns, and consistent production of uncontaminated saleable products. On this basis, a review is made, by reference to the literature, of the special characteristics of typical corrosion-resisting nickel-, cobalt- and iron-base alloys. Specific examples of economically justifiable applications are quoted. Compositions of the alloys discussed are given in table I below.

The characteristics of the respective materials are discussed, together with resulting typical applications in chemical plant. Among the points to which particular attention is drawn are the following:

Pure *molybdenum* displays unusual resistance to

Typical Corrosion-Resisting Alloys

		Ni %	Cr %	Mo %	Fe %	Co %	W %	C %	Si %	Mn %	Other Elements %
Group 1	*Alloy*										
	(1)	65	1 max.	28	5·5	—	—	0·08 max.	1 max.	1 max.	—
Available in	(2)	56	16·5	17	5·5	—	4·5	0·12 ,,	1 ,,	1 ,,	—
wrought and	(3)	45·5	22	6·5	22	—	—	0·08 ,,	1 ,,	1 ,,	Nb+Ta2
cast forms	(4)	10	20	—	2	50	15	0·10	1 ,,	1·5	—
	(5)	—	30	—	—	64	4·5	1·1	1 ,,	1 max.	—
Group 2											
Available	(6)	—	—	—	84	—	—	0·85	14·5	0·65	—
in cast	(7)	—	—	3	81	—	—	0·85	14·5	0·65	—
form only	(8)	85	—	—	—	—	—	0·12 max.	9·5	1	Cu 4

hydrochloric and hydrofluoric acids, but is rapidly attacked by oxidizing acids such as nitric and hot concentrated sulphuric acids. High-molybdenum alloys tend to show similar corrosion-resistance, e.g., *nickel-molybdenum* alloys are suitable for use in contact with hydrochloric acid and with a wide variety of non-oxidizing acids. Examples are given of equipment in which these properties are utilized. The high-nickel nickel-molybdenum alloys are suitable also for handling sulphuric acid in a wide range of concentrations. Other applications cited in connexion with such materials involve contact with halogenated organic substances or inorganic halides in organic carriers, in the pharmaceutical industry; chloride-containing catalysts, oxide slurries containing sulphuric acid, and spray nozzles handling magnesium-chloride liquors.

The oxidation-resistance imparted to many metals by *chromium* is next considered. Alloys containing chromium + molybdenum show resistance to both oxidizing and non-oxidizing environments, for example, the *nickel - molybdenum - chromium alloy* (No. 2) is one of the few metallic materials resistant to corrosion by wet chlorine gas. It also has good general resistance to ferric and cupric chlorides and other oxidizing salts, to alkalis, and to organic and mineral acids. Examples are given of its use to resist attack by pure sulphuric acid, hydrochloric acid, seawater and marine atmospheres. Reference is made also to the suitability of such alloys for use in plant in which chlorides in process streams could cause stress-corrosion cracking of stainless steels.

The *nickel-chromium-molybdenum-iron alloy* (No. 3), which is of related type, possesses the general corrosion-resisting qualities resulting from its chromium and molybdenum content, together with the economic advantage of a high iron : molybdenum ratio. This alloy has found useful application as a lining for sulphite digesters in the pulp industry, and it has the further advantage of good resistance to stress-corrosion cracking in some conditions in which austenitic chromium-nickel steels would be susceptible.

The *cobalt-base alloys* discussed are characterized by high-temperature strength (which has been exploited in such applications as turbine blades), associated with good resistance to corrosion. The wrought *cobalt-chromium-tungsten-nickel alloy* (No. 4) is reported to show good resistance to fuming nitric acid, to wet chlorine and to hydrochloric acid. The *cobalt-chromium-tungsten-carbon alloy* (No. 5), in addition to its application as a tool material, is useful where simultaneous resistance to abrasion and corrosion is required.

Silicon is known to have a significantly beneficial influence on the corrosion-resistance of iron- and nickel-base alloys and to enhance the oxidation-resistance of many alloys. A limitation on the applications of high-silicon alloys is imposed by the fact that the silicon content required for optimum corrosion- or oxidation-resistance restricts their use to the form of castings which, due to lack of ductility, are subject to failure under mechanical and thermal shock. The *iron-silicon alloys* (No. 6) which resist many severe acid conditions, are, however, widely used as pump and valve materials, and find some application as heat-resisting alloys. A *molybdenum-modified type* of such alloys (No. 7) is employed mainly in service involving contact with chlorides and hydrochloric acid.

The *nickel-silicon-copper alloy* (No. 8) is used primarily for sulphuric-acid service, but, like the iron-silicon alloys, has good resistance also to many other acid conditions, and shows high resistance to abrasion.

In a later section of the paper, consideration is given to design factors significant for ensuring optimum corrosion-resistance of the respective alloys in service. Among the points mentioned are the avoidance of sites at which corrosive media can concentrate, and the deleterious influence of galvanic coupling. The final section is devoted to discussion of the mechanical and metallurgical characteristics of the typical alloys reviewed. Properties representative of these materials are tabulated and the effect of various methods of welding is considered. Electric-arc welding, with a covered electrode, has proved to be the technique most generally suitable: oxy-acetylene welding is to be avoided, due to the possibility of diminished corrosion-resistance resulting from carbon pick-up. Precautions necessary in welding are briefly discussed, and some notes are added on fabrication procedures such as machining, strip-lining and cladding.

The paper ends with a review of the effects of various heat-treatments on the microstructure of the alloys. Mention is made of the harmful effect of sensitization, and attention is drawn to thermal-treatment conditions necessary to ensure optimum corrosion-resistance and mechanical properties in the materials as put into service. Photomicrographs of a nickel-chromium-molybdenum alloy are used to illustrate the structures produced by various thermal treatments.

Stainless-Steel Equipment in Pulp Mills

J. KOLBERG: 'Combating Corrosion in a Pulp Mill with Stainless Steel.'

Chemical Engineering Progress, 1957, vol. 53, July, pp. 353-6.

Stainless steels of the chromium-nickel type, especially the molybdenum-containing compositions, have been successfully used in the sulphite-pulp industry for some years and are being increasingly employed for replacement of equipment such as pipe lines, valves, screens and other fittings. This article reports experience which demonstrates the importance of correct choice of composition of the steel and operating conditions in mills in which especially severe conditions exist.

Abnormal corrosion, including intergranular attack, pitting, crevice corrosion and corrosion fatigue, in a large pulp plant in the U.S.A. was found to be due

by an unusually high level of chlorides in the sulphite acid and in the recovery system.

Survey of the literature on pit corrosion revealed that the frequency of pitting of stainless steels in an acid solution containing chlorides is at a minimum when the pH of the solution is between 3·5 and 4·5, and that stainless steels containing 2–4 per cent. of molybdenum are much more resistant in such conditions than steels of lower molybdenum content. In the plant where the deterioration had occurred the spent sulphite liquors entering the evaporator bodies had a pH below 3·0 and the molybdenum content of the steel of the failed tubes was only about 2 per cent. Replacement tubes having a molybdenum content of 2·27 and 2·47, per cent., on being examined after 16 months' service, showed less corrosion, and tubes made from steel in which molybdenum was 3·0 per cent. confirmed that this higher content gave even greater resistance to pitting attack. The firm therefore currently specifies for its tubes a molybdenum content of 2·75 per cent. minimum, and the carbon is required to be 0·04 to 0·06 per cent. (preferred) with a 0·07 per cent. maximum. It is also specified that the steel shall be fully annealed, and that if it has been cold-worked re-annealing is to be carried out, followed by water-quenching.

It was also found that the condensate from the evaporators was effective in passivating molybdenum-containing stainless steels. Arrangements have therefore been made for passivation of newly installed evaporator tubes by treatment with the evaporated condensate before they are placed in service involving contact with the spent liquor. In this connexion it is recorded that experimental data recently reported in the literature have indicated that resistance to pitting corrosion may be improved 15- to 30-fold by correct passivation.

The above modifications have been supplemented, in the plant described, by alterations in design calculated to prevent crevice corrosion and stress corrosion, and the welding technique used has been improved to prevent dilution of austenitic steel with carbon steel during welding.

Stainless-Steel Wire for Cold Heading

S. E. TYSON: 'Stainless-Steel Cold-Heading Wire.' *Wire and Wire Products*, 1957, vol. 32, Oct., pp. 1193–7, 1248–50.

The scope of this paper is clearly defined as covering, from a purely metallurgical standpoint, the experience of The Carpenter Steel Company, Reading, Penn., in production and processing of stainless-steel wire which meets the requirements of the cold-heading industry. The subject is dealt with in three sections, relating, respectively, to selection of the composition of the steels, wire-mill processing, and the cold-heading operation.

Choice of grade of steel is determined by a balanced consideration of the corrosion-resisting qualities of the various types, their ability to sustain the cold-heading operations without cracking or fracture, and their workability from the point of view of tool life. On the basis of these requirements, an assessment is made of the relative suitability of four grades of straight high-chromium steels of standard A.I.S.I. types, Greek Ascoloy (a 12–14 per cent. chromium steel containing small amounts of nickel and tungsten), four standard grades of austenitic chromium-nickel-(molybdenum) steel, a higher-alloy steel (Carpenter 10), Carpenter 20 (complex austenitic chromium-nickel steel containing also copper and molybdenum), and A-286 (a complex precipitation-hardening austenitic steel containing titanium and aluminium). Work-hardening characteristics (an important factor in relation to suitability for cold heading) are discussed, and the mechanical properties of the materials in the annealed condition are tabulated.

In connexion with choice of composition, attention is drawn to the importance of nickel content, as lessening the work-hardening effect during cold heading and making possible the production of cold-headed parts which are non-magnetic in the finally formed condition.

With regard to production control, strong emphasis is placed on the importance of inspection of billets for internal soundness. In wire-mill processing it has been found that, irrespective of composition, the best cold-heading wire is produced by annealing to a dead-soft condition at a size slightly larger than the finished dimension. After that operation the wire is specially coated, for lubrication, and is cold-drawn to final size. (It has been found that wire finally cold drawn works better in the later operations than material in a finally annealed condition.)

Many years of experience have demonstrated that the best general coating for the cold heading of austenitic stainless-steel wire is copper plating, followed by lubrication applied during the final drafting operation. Following plating the wire is coated with a substance such as lime, which serves as a carrier to drag the wire-drawing lubricant through the die during the final draft and provides extra separation between the wire and the header dies. Lubricants for the final draft depend on the type of material being processed and on the cold-heading operation: sodium-stearate soaps may be used for some of the straight-chromium steels, but for materials which are more difficult to head aluminium stearate or special waxes are needed.

In connexion with the cold-heading operation itself, the author makes recommendations on shearing technique, head design (including minimizing of stresses by avoidance of sharp corners), headability parameters, optimum speeds for the heading operation, etc. The importance of tool life is also considered, and in the final section of the paper some typical defects encountered in headed parts are described, as illustrating the problems involved.

Effect of Hydrogen upon Martensite Formation in Steels

See abstract on p. 9.

Extraction of Minor Phases from Chromium-Nickel Austenitic Steels

J. F. BROWN, W. D. CLARK and A. PARKER: 'The Extraction of Minor Phases from Austenitic Steel.' *Metallurgia*, 1957, vol. 56, Nov., pp. 215-23.

Investigation of phases present in alloys has become increasingly necessary as a means of interpreting properties, but techniques originally used for phase analysis (optical microscopy and thermal analysis) are inadequate for complete study of complex alloys, e.g., those used in high-temperature and chemical engineering. X-ray technique and the electron microscope have widened the range of tools of the metallurgist, but even these methods are subject to limitations with regard to constituents present in small amounts. During the past few years there has been further advance, by the application, to metals and alloys, of a modification of the technique used for isolation of non-metallic inclusions in irons and steel. The essential principle of this method is to remove the major phases present, by means of a reagent which leaves the minor phases untouched, followed by concentration of the minor phases and identification by X-ray and other techniques.

For isolation of non-metallic inclusions in steel the bulk of the sample is usually vaporized in chlorine or dissolved in alcoholic iodine; in the more recent work on austenitic steels the usual procedure has been to attack the specimen anodically, in an aqueous electrolyte. The latter method is not yet fully exploited, and suffers from certain limitations which are critically considered by the authors, but it offers considerable promise, as it is further developed.

The first part of this paper is concerned with the various techniques which have been employed for extraction of minor phases from austenitic steels and with the development of optimum apparatus and methods. Various types of extraction cell which have been used for the purpose are discussed and illustrated, and a report is made of extraction tests on three steels: see details in right-hand column.

18-8	(½ hr. 1150°C.; w.q.
18-8-Ti	(½ hr. 1150°C.; w.q.)
18-8-Ti	(½ hr. 1350°C.; w.q., +2 hr. 650°C.; w.q.)
18-8-Mo-Ti	(½ hr. 1150°C.; w.q.)
18-8-Mo-Ti	(½ hr. 1150°C.; w.q., +4 hr. 850°C.; w.q.)

Minor phases in the above steels were extracted by six methods, details of which are given in the table below.

Comparison of the efficiency of the six procedures was on the basis of the number of minor constituents preserved by each process: a detailed report is made of the results obtained on each steel with each of the six extraction procedures. (An appendix to the paper lists the characteristic spacings by which the various phases likely to be present in the extracts are normally identified on powder photographs.)

The results led to the conclusion that procedure 3 (alcoholic 10% hydrochloric acid, 0·08 amp./cm.², 40°C. max.) gives the best results for phases other than *alpha* and *gamma*. *Inter alia*, the value of the technique is demonstrated by the fact that it provided definite evidence, not obtainable by other means, of the presence of chromium carbide, and also showed the formation of *chi* phase in re-heated 18-8-Mo-Ti and indications of changes in the composition of TiC.

The second part of the report deals with comparative investigation of the structures found in 18-8-Ti and 18-8-Nb chromium-nickel steels, with particular reference to the rôle of chromium carbide in connexion with the relative corrosion-resistance of the two types of steel after re-heating. Three steels of each type (carbon approximately 1 per cent.) were softened by heating at 1050° or 1250°C. and subsequently re-heated, for periods varying between 5 minutes and 500 hours, at temperatures ranging from 500° to 900°C. Special attention was given to specimens softened at 1050°C. The heat-treated specimens were de-scaled by pickling and were then subjected to selective solution in 10 per cent. alcoholic hydrochloric acid at 0·08 amp./dm², using apparatus which is described in the paper. The extracts were examined by the techniques described in Part I, the structures of the

Table I

Conditions used for Extraction of Minor Phases in Austenitic Steels

Code	Electrolyte	Normal Current Density (amp./cm².)	Operating Temperature (°C.)	Anode Efficiency (g./amp. hr.)
1	5% w/v HCl in water	0·08	12-18	0·57-0·92
2	20% w/v HCl in water	4·7	40-80	0·70-1·0
3	10% v/v 35% aqueous HCl in ethyl alcohol	0·08	30-50	0·78-0·96
4	10% w/v H_2SO_4 in water	0·08	12-19	0·23-0·57
5	10% w/v CrO_3 in water	0·08	12-20	0·19-0·55
6	10% w/v CrO_3 in water	1·1	20-90	0·24-0·47

steels were examined, and corrosion tests were made, in 70 per cent. nitric acid, at 70°C., on each steel in the condition as air-cooled from 1050°C. and after air-cooling from that temperature followed by two hours re-heating at 650°C.

Correlation of the behaviour of the steels under corrosion test, with the structural condition after the various treatments, demonstrates that, since chromium carbide can appear in some 18-8-Nb steels, the generally acknowledged superiority of these steels to corrosion in the 'sensitized' condition is not completely and simply explicable by the theory which attributes the difference solely to the presence of chromium carbide. Such explanation requires, at least, consideration of the amount of carbide present.

Supplementary corrosion tests were made to assess the relation between rate of corrosion and change from carbide to *sigma* phase. On the results the authors make the following comments:

'The figures indicate that the liability to high corrosion rates exists only when chromium carbide is present. When heating is carried out for such a period that *sigma* replaces carbide the corrosion-resistance recovers. It is generally agreed that the effect of precipitated chromium carbide, e.g., in plain 18-8 chromium-nickel steels, is due not to the presence of the carbide, but to the depletion of the chromium content of the matrix round the carbide particles. The mechanism of the recovery of resistance requires careful consideration.

'*Sigma* contains more chromium than the basis metal. Carbide contains an even higher percentage of chromium, but the total amount of carbide which can form is strictly limited. The amount of *sigma* which can form is limited by other factors, but there is no doubt that more *sigma* might form than carbide. Hence, there is no *prima facie* reason for supposing that the change *carbide→sigma* releases chromium; it is very likely to do the reverse. The improvement in corrosion-resistance is therefore not due to the *carbide→sigma* change. There can be little doubt that the improvement is due to diffusion of chromium from the bulk of the austenite to replenish the depleted zones adjacent to the carbide particles. The sequence of events is uncertain: i.e., whether replenishment leads or follows the appearance of *sigma*. Since *sigma* formation probably involves withdrawal of extra quantities of chromium from the matrix, it is likely that *sigma* forms only after considerable levelling-out of the chromium content has taken place. This implies that the improvement in corrosion-resistance could take place while carbide was still present and before *sigma* formed.'

Among other points of interest with regard to structure are the following:

The *chi* phase was found in an austenitic steel containing only 3·2 per cent. of molybdenum.

A phase termed *tau*, observed in the 18-8-Ti steels, is shown to be probably Ti_2S.

Automatic Direct-Reading Spectrograph

See abstract on p. 2.

X-Ray Spectrographic Analysis of Nickel-base Alloys

V. W. PALEN: 'Speed Alloy Analysis by X-Ray Method.'

Iron Age, 1957, vol. 180, Oct. 17, pp. 120–1.

The article describes the procedure used in the Kokomo Plant of Haynes Stellite Company, and urges the advantages of the technique as a production-control method. The procedure described is applicable to the analysis of 29 different grades of alloy and can estimate any one of nine alloying constituents (including chromium, nickel, cobalt, titanium, tungsten, iron), in quantities ranging from 1½ to 30 wt. per cent. The alloys quoted as susceptible to such analysis are the iron-base alloys Haynes No. 93, Multimet alloy and the 300 series of nickel-chromium stainless steels, the nickel-molybdenum-(chromium) alloys Hastelloy B, C, F and X; THET alloy (complex nickel - chromium - cobalt - molybdenum - tungsten alloy), GMR 235, and cobalt-base alloys of the Haynes Stellite types. Methods of preparing the samples are described.

The spectrograph is stated to be suitable also for determination of surface depletion of elements occurring during annealing and pickling processes, and for sorting mixed welding-rod stock.

Production of Oxide-free Powders for Powder-Metallurgy Use

P. SIKORA and P. CLARKIN: 'Reduction of Oxidized Nichrome V Powders and Sintering of Nichrome V Bodies.'

Nat. Advisory Committee for Aeronautics Tech. Note 4032, Sept. 1957; 17 pp.

In view of the immense potential value of powder metallurgy, as a research tool, in high-temperature engineering, and in other applications, it is obviously desirable to build up a fund of basic information on the factors affecting the properties of powders and on the variables which influence such properties. To this end, an extensive research is being carried out at the Lewis Flight Laboratory, covering the effects, on the strength properties of high-temperature alloys produced by powder-metallurgy methods, of variations in quantity and distribution of micro-constitutents. This report deals with a preliminary phase of the work, *viz.*, standardization of techniques for producing essentially oxide-free powders and for fabricating components from such powders.

The main objectives of the work were determination of the effectiveness of various deoxidation and sintering techniques (by comparing the strengths and microstructures of powder-metallurgy specimens with those of wrought specimens), determination of the effect of residual-oxide content on the tensile properties of powder-metallurgy specimens, and evaluation, by such means, of the various methods used to produce essentially oxide-free powders.

Oxidized 80-20 nickel-chromium alloy powder, obtained from three sources, was deoxidized by three different methods of reduction; the reducing agents were, respectively, purified hydrogen, 1 per cent.

sodium hydride + purified hydrogen, and 1 per cent. carbon + purified hydrogen. Methods of treatment, and of production of compacts from the oxide-free powders, are described. Tensile specimens, prepared from representative compacts, were subjected to tensile tests at room temperature, densities were determined by the water-displacement method, and specimens were metallographically examined for oxide inclusions and the presence of voids.

The test results led to the following conclusions:

All three reduction methods were reasonably effective in lowering the oxide content of the as-received powders; the technique employing pure hydrogen and treatment in a rotating-tube furnace gave the best results, and has the advantage of ease of operation.

Several of the powder-prepared specimens showed mechanical properties and structure equivalent to those of test bars of wrought 80–20 alloy. No consistent relationship could be determined between the tensile properties of the powder-prepared specimens and their oxide contents, as revealed by oxygen analyses and metallographic examination. It was, however, found that specimens which had extremely high oxygen contents and low densities showed correspondingly low tensile strengths.

Microstructural examination indicated that the group of specimens appearing to have the highest number of oxide inclusions had the highest tensile strengths. Such inclusions were globular in form and were randomly distributed throughout the structure.

Stability of Cyanogen: Monel Containers

R. P. WELCHER, D. J. BERETS and L. E. SENTZ: 'Stability of Cyanogen.'

Industrial and Engineering Chemistry, 1957, vol. 49, Oct., pp. 1755–8.

The work described was planned to investigate reports in the literature that cyanogen is unstable and can, under certain conditions, detonate by local decomposition to its constituent elements, a possibility which was apparently supported by the fact that cyanogen has a large endothermic heat of formation and is known to be one of the most reactive nitriles. Two groups of experiments were carried out: (1) to determine the stability of cyanogen to heat, pressure and various chemical additives, (2) to investigate its reaction to mechanical shock.

Previous investigators had confirmed that, in the absence of moisture and light, gaseous cyanogen could be kept indefinitely at room temperature and atmospheric pressure. At elevated temperatures and high pressures, however, certain substances catalyze formation of paracyanogen and a hazard exists if such polymerization is initiated, for example, by trace amounts of impurities accidentally introduced into the cyanogen before storage in cylinders. Prolonged tests in evacuated sealed tubes were therefore carried out with liquid cyanogen, under its own vapour pressure, at 65°C. To establish its reaction to the presence of impurities, small quantities of salts, acids, bases, copper wire and turnings of Type 304 nickel-chromium stainless steel and of Monel were added to the cyanogen. It is concluded from the results that pure cyanogen does not polymerize or decompose at moderate temperatures; that acids, bases and salts cause a somewhat accelerated change of a small part of cyanogen, but do not induce chain polymerization or decomposition, and that pure cyanogen may be safely stored in Monel or stainless-steel cylinders without the addition of a stabilizer.

Tests in which cyanogen was subjected to severe mechanical shock showed that it was stable under such conditions and would safely withstand rough handling during shipment or other usage involving impact.

Shielding Gases for Use in Welding Nickel-containing Materials

W. H. HELMBRECHT and G. W. OYLER: 'Shielding Gases for Inert-Gas Welding.'

Welding Jnl., 1957, vol. 36, Oct., pp. 969–79.

The authors briefly outline the principles and mechanism of gas shielding, and review in some detail recent developments, with particular reference to specialized gas mixtures which have been found suitable for individual materials. The review is prefaced by a discussion of the characteristics of gases and of the properties which affect the efficacy of the protective types.

Broad recommendations are then made (mainly on the basis of metal and process) for welding of some ferrous and non-ferrous materials. It is emphasized, however, that final selection of the shielding gas best suited for any individual welding operation necessarily presumes a balanced consideration of type and thickness of material, design and fixturing, and speed and quality requirements. The materials for which specific recommendations are made are aluminium; mild steel; stainless steel and high-temperature alloys; copper, nickel and their alloys; magnesium, and titanium. The principal recommendations made for the welding of nickel-containing materials are briefly summarized below:

'*Stainless Steel and High-Temperature Alloys*

'Argon is recommended for the manual tungsten-arc welding of a wide range of thicknesses, in all positions. In a great many mechanized welding applications, such as fabrication of jet engines, food equipment and chemical apparatus, argon, argon-helium mixtures, argon-hydrogen mixtures, or helium may be used. Argon is generally preferred for welding materials up to about 14-gauge where the maximum possible speed is not required. On the heavier materials, especially where maximum speed is more important, argon-helium mixtures are preferred. In continuous welding operations such as tube welding, where speed and bead contour are of paramount importance, helium or an argon-hydrogen mixture is recommended.

'In the inert-gas consumable-electrode welding of stainless steel, argon with 1 per cent. of oxygen is generally recommended. In many applications, especially at high speeds, it is impossible to make a weld with argon because of poor coalescence, but with the addition of even 1 per cent. oxygen, excellent contour welds, fairing smoothly into the base plate, can be produced. Helium or argon-helium mixtures also are not recommended, since without the addition of oxygen they give poor coalescence, as well as poor arc characteristics and spatter. Argon with 5 per cent. oxygen is recommended in those applications, especially under $\frac{3}{16}$ in., where the addition of 1 per cent. oxygen does not give the desired arc stability, coalescence or welding speed. The use of 5 per cent. oxygen will result in slight but harmless discoloration or oxidation of the weld-metal surface. Microexamination of welds made under argon with 5 per cent. oxygen shows them to be sound and free of inclusions: the tensile, yield, elongation and impact properties are satisfactory, and corrosion-resistance of welds so made is of a high order.

'*Copper, Nickel and Their Alloys*

'Argon is recommended for the manual tungsten-arc welding of copper, nickel and copper-nickel alloys. Argon-helium mixtures have some advantage in the welding of the heavier thicknesses. Helium is preferred for welding heavy sections, because the increased voltage provides a hotter arc for welding these high-melting-point, high-conductivity metals. Argon can be employed if auxiliary pre-heat is used.

'In mechanized operations argon is preferred for welding these materials up to the thickness of about $\frac{1}{16}$ in. For all applications involving greater thicknesses helium or an argon-helium mixture is recommended.

'In the mechanized inert-gas-shielded consumable-electrode welding of copper, nickel and their alloys, argon is preferred for welding material up to $\frac{1}{8}$ in. in thickness. For the welding of thicker plate an argon-helium mixture is preferred. The amount of helium in this mixture varies between 50 and 75 per cent., depending on the type of joint and the thickness of material employed. (Examples of preferred shielding gases for welding two thicknesses of Monel and Inconel are tabulated in the paper.) In the case of welding Monel and Inconel alloys in the $\frac{1}{4}$-in. double-vee joint, a choice can be made between pure argon and a 50-50 argon-helium mixture, since the penetrating characteristic of helium is not as essential in a bevelled joint as in a square butt joint. Helium should not be used for consumable-electrode welding of these metals because of the inherent arc instability and evolution of spatter.'

The article closes with notes on some ternary argon-helium-oxygen mixtures which have recently been experimentally developed, but which, due to problems of mixing and supply, have not yet been commercially promoted. Such mixtures have been effectively used, *inter alia*, in the inert-gas-shielded consumable-electrode process for the welding of stainless steel. Reference is also made to tentative work on argon/carbon-dioxide mixtures, and to argon-nitrogen and argon-chlorine types.

Crack Sensitivity of Austenitic Welds: Influence of Composition

A. HOERL and T. J. MOORE: 'The Welding of Type 347 Steel. Statistical Analysis of the Effects of Various Elements on the Crack Sensitivity of Fully Austenitic Type 347 Welds.'

Welding Jnl., 1957, vol. 36, Oct., pp. 442S-8S.

The material contained in this paper is substantially identical with that reported in *U.S. Atomic Energy Commission Publication NYO-3500*: see abstract in *Nickel Bulletin*, 1957, vol. 30, No. 10, p. 193.

Stress Relief in Weldments

See abstract on p. 2.

Brazing of Aluminium Bronze to Inconel

D. CAPLAN and E. F. NIPPES: 'High-Temperature Brazing of Aluminium Bronze to Inconel.'

Welding Jnl., 1957, vol. 36, Oct., pp. 457S-64S.

A composite suitable for a heat exchanger operating in air at temperatures as high as 1500°F. (815°C.) is one consisting of aluminium-bronze fins joined to Inconel tubes. The bronze approaches the ideal as a fin material, since it has both the high thermal conductivity of copper and the good oxidation-resistance resulting from the aluminium-oxide barrier film which forms on its surface. Inconel as the tube material is characterized by good high-temperature strength, oxidation-resistance, and resistance to corrosion by liquid-metal heat-exchange fluids. A preferred method of fabrication is by brazing, using a nickel-chromium-base high-temperature brazing alloy, but a problem arises because the aluminium-oxide film which gives the bronze its oxidation-resistance interferes with the wetting of the alloy by the brazing material. Use of a suitable flux may be expected to promote brazing, but the design of a heat exchanger comprises inaccessible joints which make flux removal a major problem, and corrosion failure is likely to occur in service. A further complication is that the solidus temperature of the aluminium bronze is unduly close to the flow temperature of the filler metals.

The purpose of the investigation described in this paper was to determine the conditions in which it would be possible to braze aluminium bronze without a flux, or, alternatively, to develop a satisfactory flux sufficiently soluble to be easily removed.

It was found that in spite of the film of alumina, brazing of aluminium bronze to Inconel in high-purity hydrogen is possible, if precautions are taken to minimize the thickness of the film. Essential conditions are a rapid rate of heating, and rapid flow of high-purity hydrogen to sweep away oxygen resulting from the dissociation of the chromium oxide on the Inconel. While such precautions are possible in laboratory work, they are not practicable in large-scale production.

Fluxes based on borax are effective, but are undesirable due to the limited solubility of the flux residues, which would be difficult to remove from an intricate heat exchanger. Chloride-based fluxes are also effective and have the advantage of leaving residues of a more soluble character. A preferred mixture is 5 parts of sodium chloride, 2 parts of lithium chloride and 1 part of copper chloride. A nickel-base brazing alloy containing boron, silicon and iron (and, optionally, chromium) is suitable.

The mechanism of fluxing with chloride fluxes appears to depend on an 'undermining' of the oxide film resulting from operation of the electrochemical cell Al_2O_3/flux/bronze. Formation of complex oxides of the form $LiAlO_2$, which are soluble in the molten salt, either contributes to the undermining effect or is an alternative mechanism.

PATENTS

Electron-Emissive Cathode for Magnetrons

A magnetron cathode structure consists of a molybdenum core with an electron-emissive coating in the form of a sintered matrix of nickel-coated tungsten particles interspersed with electron-emissive material (preferably one or more alkaline-earth oxides). The ratio of nickel on the tungsten particles to the amount of tungsten (4 : 1) is such that the thermal expansion of the coating is the same as that of the core. Claims are made also for the method by which the electron-emissive coating is fabricated.

WESTERN ELECTRIC CO., INC.
Brit. Pat. 783,836.

Bright Nickel-Plating Solutions

Electrolytes for use in deposition of nickel are of nickel-sulphate and/or nickel-chloride base and contain three brightening agents: (1) of formula $(C_6H_5SO_2NHSO_2C_6H_4)_2$, or a compound incorporating such a radical, (2) a naphthalene sulphonic acid or its salts, and (3) amino polyaryl amines or methanes. The preferred basis solutions to which these additions may be made are of the following types:

	g./L.
$NiSO_4.7H_2O$	100–400 (preferably 200–300)
$NiCl_26H_2O$	10–60 (preferably 25–40)
H_3BO_3	0–50 (preferably 15–40)
H_2O to make	1000 c.c.

Temperature 100°–160° F. (38°–70° C.)
preferably 120°–140° F. (50°–60° C.)
pH 3·5–5 (preferably 4·0–4·5)
Cathode current density 30–60 amp./sq. ft.

HARSHAW CHEMICAL CO. *Brit. Pat.* 785,931.

Bright-Nickel-Plating Solutions

Bright nickel deposits, which are stated to have the added advantages of ductility and low internal stress, are produced from nickel-plating solutions of various basis types, using indazole derivatives as brightening agents. The compositions of typical electrolytes are embodied in the specification.

METAL AND THERMIT CORPN. (inventor, F. PASSAL).
Brit. Pat. 784,139.

Nickel Plating of Oxidized Parts

Parts on which an oxide film is present, e.g., aluminium or chromium, may be plated by the following treatment:

After degreasing, the surface is etched, and then treated in a bath the current density of which is initially maintained at a level below the minimum value which would cause incipient electrodeposition and then slowly increased to reach normal electrodeposition value. In the initial treatment evolution of hydrogen from the electrolyte is necessary in order effectively to reduce the oxide layer. The following details are given of a typical nickel-plating bath:

	g./L.
$NiSO_4.6H_2O$	140
$Na_2SO_4.10H_2O$	150
NaCl	24
H_3BO_3	30
Free H_2SO_4	4-9

Operating temperature may be 32° C., and the current density for the final plating is 10 amp./sq. dm.

BOZEL – MALETRA, STE. INDUSTRIELLE DE PRODUITS CHIMIQUES. *Brit. Pat.* 785,503.

Maintenance of Electroless Plating Solutions

A plating bath consisting of nickel ions and hypophosphite ions is regenerated by adding nickel hypophosphite and $H(H_2PO_2)$ or alkaline hypophosphites and soluble alkali, to minimize introduction of extraneous ions and the building up of undesirable salts.

GENERAL AMERICAN TRANSPORTATION CORPN.
(inventor, P. TALMEY).
Brit. Pat. 786,174.
(Similar to *U.S. Pat.* 2,766,138.)

Electroless Nickel Plating

A series of patents recently published claims improvements in composition of solutions, maintenance technique, preparatory treatment of the basis metals, and means by which thicker deposits of a satisfactory character can be obtained. The inventions are designed specifically to facilitate batch operation of the electroless (catalytically generated) process on a commercial scale. The inventions disclosed in these patents are covered in the U.S.A., and closely similar (but not necessarily identical) patents have been published in a number of European countries, and in Australia, South Africa, India and Japan.

GENERAL AMERICAN TRANSPORTATION CORPN.
Brit. Pats. 785,693–7, 786,174 and 5.

Nickel-Silicon-Boron Alloys

Nickel-silicon-boron alloys are claimed to be particularly well suited for application as hard facings, since they are capable of being plastically formed at relatively low temperatures (e.g., over the range 150°-350°F.: 65°-180°C.). The composition range cited is silicon 2·5-5·5, boron 0·75-5·5, per cent., balance nickel, with the possible inclusion of iron and/or manganese and/or chromium in a total not more than 5 per cent. Alloys of this type are resistant to oxidation up to their melting points, and these are sufficiently low to make it easily possible to apply the alloys as facings. The alloys adhere well to stainless steel and molybdenum, they are suitable for joining strips of stainless steel and molybdenum, and are capable of being bonded with most base metals. They are particularly suitable for such applications as the facing of poppet valves.

CAST METALS, INC. (inventor, A. T. CAPE).
Brit. Pat. 785, 416.

Boron-containing Nickel-Chromium-Cobalt Alloys

Strength and scaling-resistance of nickel-chromium-cobalt-base alloys, in the range 600°-1000°C., are improved by the addition of boron, with or without molybdenum or tungsten. Tungsten, if present, should be in the range 8-15 per cent. Titanium and aluminium are included as strengthening agents. The compositional limits of the alloys, (which are suitable for both forgings and castings), are as follows: carbon 0·001-0·15, silicon 0·1-1·0, manganese 0·1-1·0, cobalt 10-25, chromium 15-25, tungsten 0 or 8-15, boron 0·001-0·05, molybdenum 0-10, titanium 1·6-2·75, aluminium 0·2-2, iron 0-5, per cent., nickel balance. Not less than 40 per cent. niobium, tantalum and vanadium, singly or together, may be added, up to a total combined content of 3 per cent. and the alloys may also incorporate up to 3 per cent. of one or more deoxidizing elements such as silicon, magnesium, calcium, cerium and zirconium.

W. JESSOP AND SONS, LTD. (inventors, G. T. HARRIS and H. C. CHILD).
Brit. Pat. 783,955 (Similar to *Belgian Pat.* 532,000 and *French Pat.* 1,106,620).

Boron + Molybdenum in Nickel-Chromium-Iron Alloys

Nickel-chromium-iron alloys of relatively high iron content are difficult to forge and therefore give some trouble in hot working for turbine parts and other components. The invention covered by this patent is based on the discovery that good forgeability, combined with high resistance to creep at temperatures up to 750°C., can be obtained in alloys of fairly high iron content if critically balanced amounts of boron and molybdenum are present. The heat-treated alloys are also characterized by good ductility throughout the range room temperature to 750°C. The basis alloys are of the well known precipitation-hardening type containing the $Ni_3(Ti,Al)$ phase. The limits of composition are claimed as nickel 25-50, chromium 8-25, carbon up to 0·2, free titanium 1·5-6, molybdenum 5-7, boron 0·03-0·15, zirconium 0-0·2, aluminium up to 4, manganese 0-3, silicon 0-1, copper 0-5, vanadium 0-1, cobalt 0-5, per cent., balance (except for impurities) iron, which is at least 20 per cent. Compositions and rupture-life values are quoted for various typical alloys and the preferred content and functions of the respective constituents are discussed. Nickel is preferably 34-45 and chromium 12-16 per cent., with low carbon. Optimum level for free titanium is 2-3 per cent. Heat-treatment is also considered in the specification, but is not part of the claims.

MOND NICKEL CO., LTD. *Brit. Pat.* 785,271.

Cerium Additions to High-Temperature Alloys

Rupture strength and forgeability of high-temperature alloys are improved by the addition of cerium in amounts up to and including 1 per cent. The modification is claimed in relation to parent alloys covered by two earlier patents (*Brit. Pats.* 674,021 and 686,180). The cerium may be added in the form of ferrocerium, or Mischmetall, as mixed rare-earth oxides, or *viâ* any alloy rich in cerium.

Both parent patents cover complex alloys of high cobalt content, incorporating also nickel, chromium, iron, molybdenum, vanadium and niobium, and, in one case, boron.

W. JESSOP AND SONS, LTD.
(inventors, G. T. HARRIS and H. C. CHILD).
Brit. Pats. 787,636 (addition to 674,021);
787,637 („ „ 686,180).

Steels for High-Temperature Steam Plant

Properties required in austenitic steels for handling steam and other parts working at elevated temperatures and at high pressures, e.g., for superheater tubes, are obtained by the use of compositions balanced with respect to strengthening elements such as tungsten, molybdenum, niobium (or niobium + tantalum), chromium, nickel, carbon and nitrogen. Proportions are such as to provide a single-phase austenitic structure permitting the forming operations involved in manufacture. After fabrication, the steels are solution treated at 2150°-2250°F. (1205°-1230°C.), followed by quenching. Such steels develop strength properties not attainable in conventional austenitic chromium-nickel steels, and are characterized also by high resistance to oxidation. The steels, which are claimed to be suitable for use at temperatures of 1100°-1500°F. (595°-815°C.), are within the following limits of composition: chromium 13·50-18·00, nickel 13·50-16·50, molybdenum 1·00-2·00, tungsten 1·00-2·00, niobium and/or tantalum 0·50-1·50, carbon 0·06-0·18, nitrogen 0·15 max., silicon 2·75 max., manganese 2·00 max., per cent. Numerous examples are given of the properties of steels within this range, in comparison with those of less complex austenitic steels.

BABCOCK AND WILCOX CO. *Brit. Pat.* 785,380.

Steels for Superheater Tubes

Forgeable austenitic steels possessing high strength, good resistance to corrosion, and freedom from susceptibility to impact embrittlement at elevated temperatures contain chromium 15–20, nickel 12–18, carbon 0·02–0·15, manganese 0·25–2·5, silicon 0·1–1, niobium+tantalum 1–3·5, copper 2–3, per cent., iron balance. Such materials are stated to be particularly suitable for superheater tubes.

F. T. EBERLE, assignor to BABCOCK AND WILCOX CO. *U.S. Pat.* 2,797,992.

Coolants for Use in Machining Nickel-Chromium-base Alloys

In machining high-temperature alloys, e.g., Nimonic alloys, using carbon dioxide as coolant, the medium is applied in such a way that only the cutting tip or edge is cooled, not the entire workpiece. By this means, it is possible to achieve cutting speeds of 0·250 × 0·015 in., at 500–1000 ft./min.

NAT. RESEARCH DEVELOPMENT CORPN. (inventors, A. R. TOMLINSON and W. S. HOLLIS). *Brit. Pat.* 784,504.

Heat-Treatment of Nickel-base Casting Alloy

The hot-working properties of a nickel-base casting alloy of the composition shown below are improved by heating at 1900°–2300°F. (1040°–1260°C.) for at least one hour per inch of cross section. Composition of alloys claimed to be susceptible to improvement by such treatment is as follows: carbon 0–0·35, silicon 0–1, manganese 0–1, chromium 10–20, molybdenum 2–24, titanium 1–4, aluminium 1–5, iron 0·01–18, boron 0·01–0·5, per cent., nickel balance.

D. K. HANINK, assignor to GENERAL MOTORS CORPN. *U.S. Pat.* 2,798,827.

Processing of Nickel-containing Watchspring Alloys

Watchsprings are produced from ferritic alloys of body-centred-cubic structure by homogenizing, cold-rolling to effect reduction by at least 80 per cent., to produce predominant (110) orientation, cutting, and heat-treatment in the range 200°–600°C. Among the alloys which can be so processed is the following composition: chromium 2–30, nickel 2–24, manganese 0·1–2, silicon 0·1–1, carbon 0·01–0·2, molybdenum 0–5, niobium 0–1, titanium 0–1, beryllium 0–1, per cent., remainder iron.

R. STRAUMANN. *Brit. Pat.* 785,004.

See also *Brit. Pat.* 784,661.

Welding of Ferritic to Austenitic Tubes

Tubes of ferritic steel may be joined to austenitic-steel tubes by means of an austenitic fusion-welding alloy having (1) an allowable working-stress characteristic substantially in excess of that of the ferritic steel member at the service temperature of the welded part, and (2) a coefficient of thermal expansion identical or substantially identical with that of the ferritic alloy-steel member. The weld metal is preferably an alloy containing at least 50 per cent. of nickel and 10 per cent. of chromium, balance iron with additions or usual impurities which do not significantly change the coefficient of thermal expansion of the alloy.

BABCOCK AND WILCOX, LTD. *Brit. Pat.* 786,604.

Heat- and Corrosion-Resisting Coatings on Metallic Materials

Heat- and corrosion-resistant coatings are applied to metal surfaces by mixing small particles of aluminium, iron, nickel or cobalt with alumina, magnesium oxide or zirconium oxide, sintering the mixture, reducing, and flame-spraying the resulting substances onto the basis surface at a temperature of at least 2100°F. (1150°C.).

E. T. MONTGOMERY, A. P. WELCH and J. L. BITONTE, assignors to OHIO STATE UNIVERSITY RESEARCH FOUNDATION. *U.S. Pat.* 2,775,531.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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